

**POSITIVE PHOTORESIST AS A
SACRIFICIAL LAYER FOR MEMS MICRO-
COMPONENT FABRICATION WITH SU-8
POLYMER**



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ABSTRACT

SU-8, a type of epoxy polymer, is a new UV-curable material for constructing micromechanical components such as those in micro-electro mechanical systems (MEMS) with high aspect ratios. This polymer is biocompatible and therefore suitable for both in-vitro/in-vivo applications. It also possesses good mechanical properties such as hardness and Young's modulus. In addition, compared to other polymers, SU-8 has other capabilities such as photosensitivity and transparency to visible light which make SU-8 compatible with micro-fabrication processes. This is a promising structural material for producing novel devices used in MEMS and bio-related applications such as drug delivery system, bio-diagnostic testing kit, bio-MEMS, micro-fluidics and other health products.

Despite the promising applications, the fabrication of SU-8 components still requires expensive steps of lithography. One such step is the lift-off process which requires metallization of the silicon substrate before SU-8 deposition and etching out of this metal layer before the release (lift-off) of the device. The process is time-consuming, expensive and often deteriorates the SU-8 surface itself because of the strong etchant and heat used during lift-off. The existing method requires a sacrificial layer of metal such as aluminium. As a result, acidic etchants are needed for the process of lift-off which etch-out the metal layer. And at the same time, heat will be required to speed up the etching process. Concentrated acid mixture such as piranha solution used as the etchant can cause severe damage to the SU-8 layer itself. In this work, we demonstrate a method to fabricate SU-8 micro-components using a novel

lift-off technique. The important aspect of the current novel method is that the photoresist AZ4620, a polymer, is used as the sacrificial layer instead of a metal layer. AZ4620 can be easily undercut by SU-8 developer and thus reducing the lift-off time considerably. Further, the silicon substrate is metallized with aluminium to reduce the surface energy and drastically shorten the AZ4620 lift-off time. This metal layer is not the sacrificial layer and hence can be reused making the whole process very time-effective and cost-effective with better SU-8 surface qualities.

Publications from this thesis:

1. "Comprehensive High Aspect- ratio Micro-structure fabrication Procedure using SU-8/nano-composite polymers (CHAMPS)" - United States Patent Application US Provisional Application No.: 61/390,222 filed on October 6, 2010.
2. Kia Hian Lau, Archit Giridhar, Sekar Hari Krishnan, Nalam Satyanarayana and Sujeet Kumar Sinha, "Releasing high aspect ratio SU-8 microstructures using AZ photoresist as a sacrificial layer on metallized Si substrate" Submitted for publication in "*Microsystem Technologies*"

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CHAPTER 1 – INTRODUCTION

1.1 BACKGROUND

Micro Electro-Mechanical System (MEMS) is a technology generally requiring high-aspect ratio micro-components with well-controlled mechanical properties to perform different applications such as motion sensing, resonating, actuation etc. The components and devices constructed so far include micro-reservoir, micro-pumps, cantilever, rotors, channels, valves and sensors. Size of the devices fabricated range between few millimeters to sub-micrometers. It can be operated either in the form of passive (a device that does not require a source of energy for its operation) or discrete (a device that requires a source of energy for its operation) mode depending on the application requirements. In order to fabricate MEMS devices, conventional method is to make use of the existing semiconductor fabrication techniques which is normally used to manufacture electronic integrated circuits. Those techniques include wet etching using either acidic or alkaline etchant, dry etching making use of reactive gases and electro-discharge machining (EDM) and other technologies capable of producing small devices. Silicon is chosen as the material for constructing MEMS devices because most of the processes are related to existing integrated circuit fabrication. Initially, silicon was considered as MEMS material due to familiarity in semiconductor processing. Later, researchers started to explore other materials such as polymers for MEMS fabrication in order to replace silicon due to its certain drawbacks such as bio-incompatibility, brittleness and expensive processing steps.

As the interest for MEMS devices to operate in-vitro/in-vivo environment are becoming more popular, fabrication methodology need to be modified for example hermetically sealing technique. At the same time, material used must be biocompatible in order to implant into the human body and the production must be cost-effective. As a result, the MEMS technology is further branching out to bio-related sector known as BioMEMS. Drug delivery system is developed from this particular technology and it has digitalized sequential control which can be well achieved with polymer based platform. Additionally, other functions such as optical, chemical sensing and electrical capability are being implemented into the system and at the same time tuned with respect to changes in the physical surrounding environment.

An important material has emerged in MEMS manufacturing and it has been used intensively over the last few years. This material is SU-8 which is a negative tone, chemically amplified, near UV photoresist. It was developed for microelectronics industry in the late 1980s by IBM as a negative photo resist for high resolution patterning which was further probed for its ability to make high-aspect ratio moulds used in LIGA process for electroplating procedures [1-2]. This type of polymeric material is rapidly replacing silicon as the next generation of MEMS material [1-4]. Unlike silicon, SU-8 is somewhat hydrophobic in nature and biocompatible [3-6]. Furthermore, it can also be used to fabricate into micro/nanostructures [3-6] with great convenience. It is a low cost acquiescent material allowing the designer to create structures defined by a number of in-plane and out-of-plane geometries which exhibit the ability to fabricate three-dimensional

structures incorporated with good mechanical properties. This versatile material has adequate physical, chemical and mechanical properties such as higher coefficient of thermal expansion, low Young's modulus, good chemical/corrosive resistance, thermal stability that favour the construction of complex 3D structures [7-8] and hierarchical patterns [9] with cost-effective fabrication procedures such as UV exposure, spin coating and developing. However, the cost of fabrication may still be high unless the processing steps are simplified. Thus, in this thesis a novel approach to fabricating SU-8 microstructure is presented. With this approach, it is possible to fabricate high aspect ratio micron- to millimetre-sized components with much cost-effective processing steps than those necessary in the current silicon process.

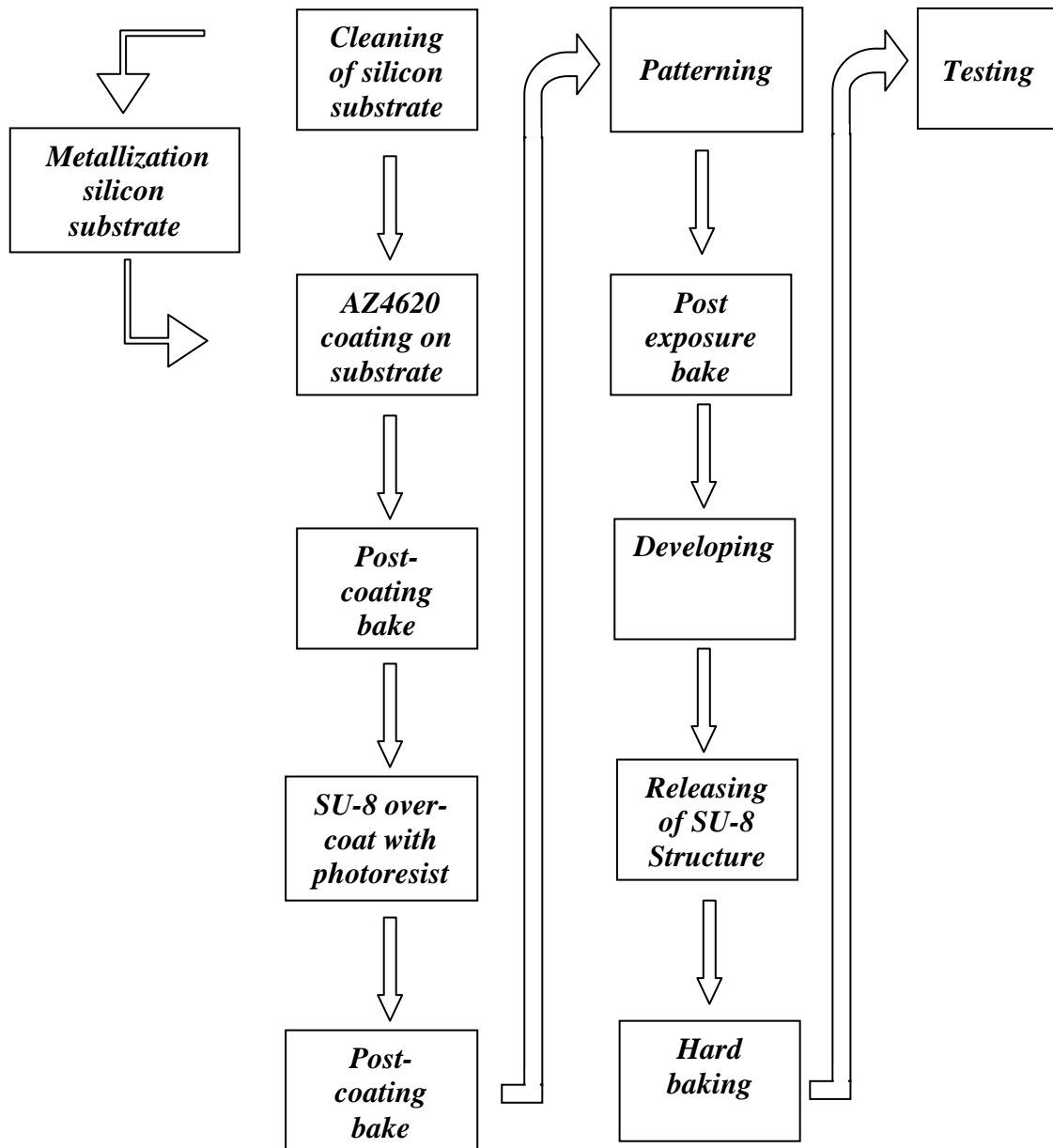
1.2 OBJECTIVES

The aim of this project is to introduce a new method of SU-8 structure lift-off from the silicon substrate such that the lift-off time is drastically reduced with enhanced surface quality requiring simpler processing steps. The project is divided into several phases as shown below:

- First phase of the project is to develop SU-8 structure and release the structure using the new lift-off technique. Mechanical testing such as indentation and tribological analysis are also carried out on the fabricated SU-8 structures.
- Second phase of the project is to further characterize the structure releasing technique in terms of the duration of lift-off taken and the amount of releasing material used in order to reduce the wastage. This also includes the application of a metallic layer on the silicon substrate that facilitates easy lift-off.
- Finally, the last phase of the project is to create micro-tips using this new SU-8 lift-off method.

1.3 PROCESS DETAILS

Following is a schematic of the process steps used in this novel SU-8 lift-off method. Some details of the procedures and tests are also presented.



CHAPTER 2 - LITERATURE REVIEW

2.1 OVERVIEW OF POLYMERS – SU-8 USED IN MEMS/BIOMEMS APPLICATION

2.1.1 CHEMICAL AND PHYSICAL PROPERTIES OF SU-8

SU-8 is an epoxy based negative photoresist which is highly functional, optically transparent having UV-curable property, biocompatible [10] and with cost-effective fabrication advantages. Once a cured film or a microstructure is fabricated, it will have resistance to chemicals at an acceptable level. At the same time, it is thermally and mechanically stable. This type of resist is normally very viscous, and as a result, it can be spread in spin coating with different thickness ranges. The thickness is dependent on the original viscosity of SU-8 produced by the manufacturer, the spinning speed of the spin coater and the amount of polymer poured onto the surface of the substrate. Further, the structure is formed by standard contact lithography technique. Figure 2.1 shows the molecule layer of SU-8. Homogenising curing process will enhance the uniformity of film properties.

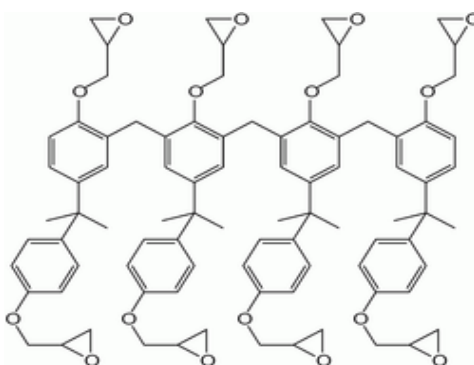


Figure 2.1: SU-8 molecule formation

2.1.2 TECHNIQUES USED FOR FABRICATION AND APPLICATION

In order to achieve mass production capability, direct LIGA is being used. LIGA means – German acronym for lithography, electroplating and moulding. Figure 2.2 show the standard process flow for LIGA process. LIGA process provides high aspect ratio micro structures in polymers e.g. PMMA (better known as acrylic glass). Via electroplating, these structures can be replicated in metals like gold, nickel, magnetic nickel-iron alloys or copper. Even replications in ceramics are possible. An industrial low cost production of micro structures is possible when a nickel tool is fabricated for hot embossing or injection moulding. The fabrication work done in this project uses the method of high aspect ratio fabrication technique to create micro devices similar to those produce by LIGA process.

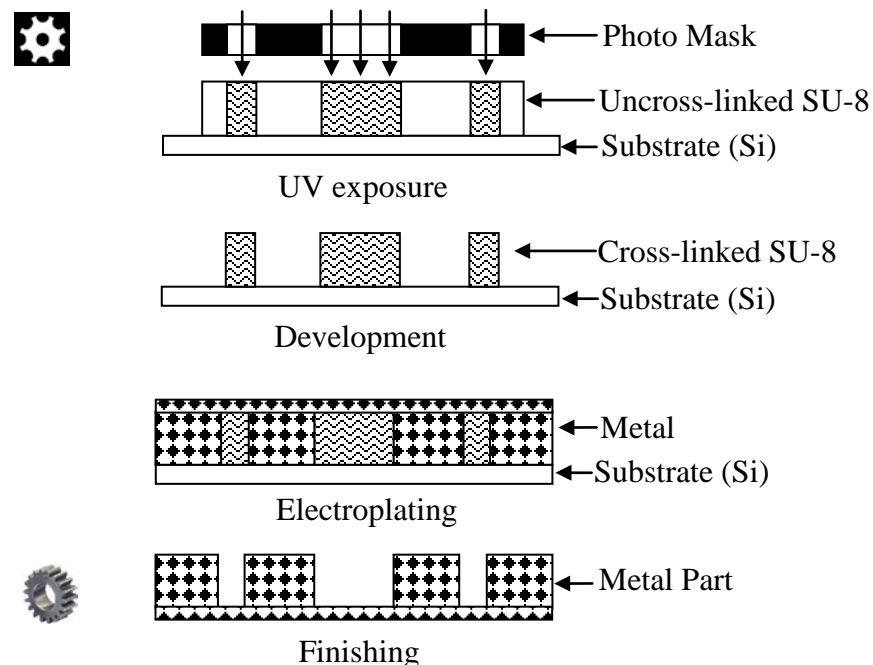


Figure 2.2: Process flow of LIGA

Table 2.1 shows some typical applications which are constructed by SU-8 developed in LIGA process. All the applications shown are commercially developed.

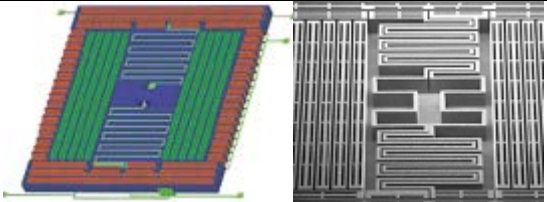
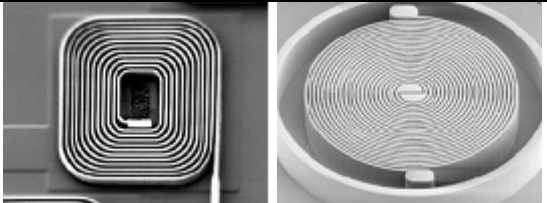
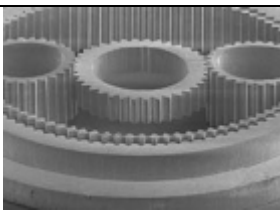
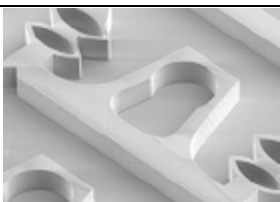
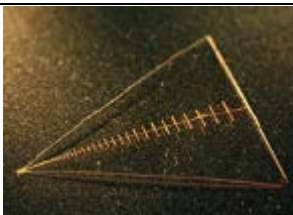

<i>Field of application</i>	<i>Application description</i>	<i>Actual device</i>
Sensors	Capacitive acceleration sensor with thickness of 200 μ m and feature size of 20 μ m which is fabricated by electrochemical deposition.	
Actuators	SU-8 able to offer the realization of high aspect ratios of conducting line for the fabrication of electro-magnetic actuator array.	
Direct LIGA	Fabrication of micro-gear and mixer for fluidic system using LIGA process. As SU-8 gives excellent sensitivity and achievable vertical side wall.	
Plastic Micro-Parts	SU-8 has special advantage for fabricating micro parts directly in synthetic material.	
Packaging	SU-8 allow application such as packaging and housing solution for electronic and sensor micro components as it sealing ability.	
Wave Guides	Chemical modification of SU-8 give rise to micro-optical wave guides device owing to changes in refractive indices.	

Table 2.1: Field application of SU-8

2.2 LIST OF RESERACH APPLICATION USING SU-8

2.2.1 NANO-INDENTATION RESULTS ON SU-8

Al-Halhoul et al. conducted mechanical property study on SU-8 using the method of nanoindentation [11]. Nanoindentation testing method has become a popular tool for characterizing polymeric materials mechanical properties, as viscoelastic-plasticity behaviour naturally inherent in polymeric materials, Young's modulus and hardness for very thin layers can be extracted from load-displacement data [12]. In order for the indentation testing to be carried out, two samples were fabricated by spin coating method on glass substrate and the thickness of SU-8 coated was 385 μm with 2 mm in width and 5 mm in length. The group carried out the nanomechanical testing with methods of quasi-static and dynamic measurements using diamond Berkovich shaped indenter tip on a triboindenter system [Figure 2.3]. From the test conducted, average values for Young's modulus, hardness, storage modulus and loss modulus were obtained. Measurement result of Young's modulus and hardness showed that the data are very close to macroscale testing methods. It is concluded that SU-8 photoresist has moderate viscoelastic behaviour and it is a promising candidate for many MEMS applications including micro-cantilevers, micro-channels and micro-molds. Table 2.2 show the results obtained from the tests conducted using indentation method on SU-8 film.

<i>Indentation Force (μN)</i>	<i>Reduced modulus (GPa)</i>	<i>Hardness (GPa)</i>
1500	5.5	0.41
3000	5.7	0.39
4500	5.8	0.38
6000	6	0.42
7500	6.1	0.46
9000	6.2	0.49

Table 2.2: Indentation result on SU-8 film

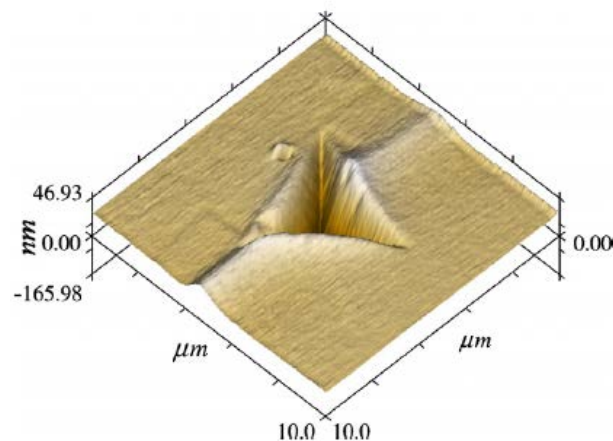


Figure 2.3: Berkovich indentation mark on SU-8 surface

2.2.2 TRIBOLOGICAL ANALYSIS STUDY

There are a few studies conducted on SU-8 with respect to tribology, *Tay et al* [13] conducted tribological study on SU-8 micro dot. Micro dots have the size approximately 100 μm in diameter fabricated by polymer jet printing technique on silicon wafer with an area of 7 x 7 mm^2 . Figure 2.4 (a) shows the schematic of micro dots on silicon wafer and Figure 2.4 (b) show the topography images of the micro dots.

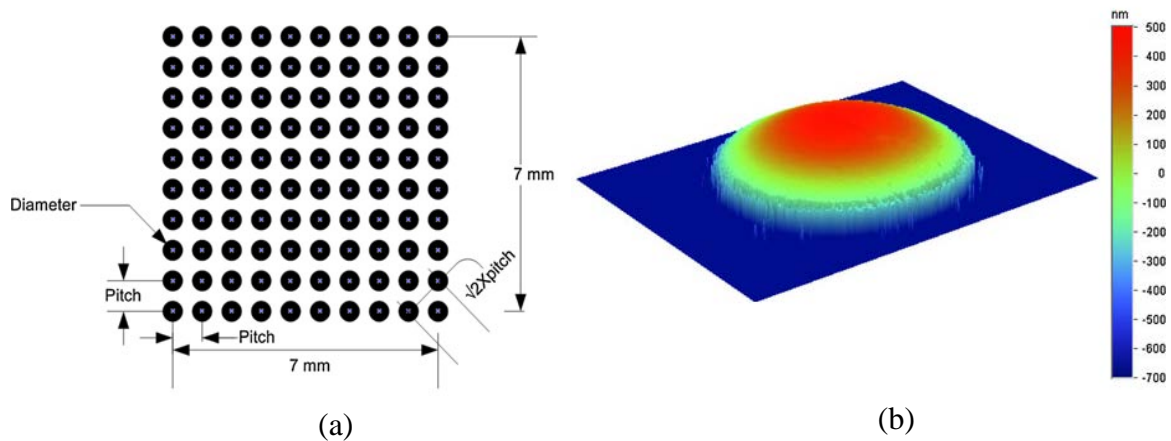


Figure 2.4: Schematic design of micro dots on silicon wafer (a) and topography images of the micro dots (b)

The results obtained from friction and wear tests, which were performed on the micro-dot pattern, show that SU-8 has lower wear life. However, Perfluoropolyether (PFPE) over-coated on SU-8 micro-dots show that there are much improvement on the wear life. Also, there is an optimum pitch between the micro-dots that would give the maximum wear life

R A Singh et al [15] conducted study with the aim of improving the tribological performance of SU-8. Experiments were setup by coating two different thickness of SU-8, 500 nm and 50 μm on the silicon wafer. Table 2.3 shows the tested materials and nomenclature used.

Material	Nomenclature	Material	Nomenclature
Bare Silicon	Si	SU8 thick film ($\sim 50 \mu\text{m}$) on Si wafer	SU8 ThF
Si coated with PFPE	Si/PFPE	SU8 thick film coated with PFPE	SU8 ThF/PFPE
Si coated with SU8 thin film ($\sim 500 \text{ nm}$)	Si/SU8 TF	SU8 thick film treated with O_2 plasma at 25 W	SU8 ThF-O2-25W
Si coated with SU8 thin film and PFPE	Si/SU8 TF/PFPE	SU8 thick film treated with O_2 plasma at 50 W	SU8 ThF-O2-50W
Si coated with SU8 thin film and treated with O_2 plasma at 50 W	Si/SU8 TF-O2-50W	SU8 thick film treated with O_2 plasma at 100 W	SU8 ThF-O2-100W
Si coated with SU8 thin film and treated with O_2 plasma at 100 W	Si/SU8 TF-O2-100W	SU8 thick film treated with O_2 plasma at 25 W and coated with PFPE	SU8 ThF-O2-25W/ PFPE
Si coated with SU8 thin film and treated with O_2 plasma at 50 W and coated with PFPE	Si/SU8 TF-O2-50W/PFPE	SU8 thick film treated with O_2 plasma at 50 W and coated with PFPE	SU8 ThF-O2-50W/PFPE
Si coated with SU8 thin film and treated with O_2 plasma at 100 W and coated with PFPE	Si/SU8 TF-O2-100W/PFPE	SU8 thick film treated with O_2 plasma at 100 W and coated with PFPE	SU8 ThF-O2-100W/ PFPE

Table 2.3: Tested material and nomenclature used

Surface characterization test were carried out in order to obtain information on water contact angle (WCA), nanoscale roughness (R_a) and material properties such as hardness and elastic modulus by nanoindentation. Table 2.2 shows the surface properties of the tested material.

Material	WCA (deg)	R_a (nm)	Material	WCA (deg)	R_a (nm)	E (GPa)	H (GPa)
Si	12	0.20	SU8 ThF	94	0.26	4.0	0.35
Si/PFPE	66	0.60	SU8 ThF/PFPE	100	0.29	4.0	0.36
Si/SU8 TF	75	0.20	SU8 ThF-O2-25W	~ 6	4.87	4.1	0.36
Si/SU8 TF/PFPE	87	0.21	SU8 ThF-O2-50W	~ 5	9.13	4.2	0.31
Si/SU8 TF-O2-50W	<5	4.80	SU8 ThF-O2-100W	<5	12.86	3.2	0.17
Si/SU8 TF-O2-100W	<5	9.23	SU8 ThF-O2-25W/PFPE	59	4.65	4.1	0.36
Si/SU8 TF-O2-50W/PFPE	79	6.80	SU8 ThF-O2-50W/PFPE	68	7.01	3.8	0.28
Si/SU8 TF-O2-100W/PFPE	92	15.60	SU8 ThF-O2-100W/ PFPE	79	8.22	3.0	0.16

Table 2.4: Surface properties of tested material

The tribological results are summarized in Table 2.4. It is seen that a suitable oxygen plasma treatment of SU-8 followed by an overcoat of PFPE gives an excellent protection against wear for SU-8.

2.2.3 FABRICATED SU-8 DEVICE FOR STRESS MEASUREMENT

There are many research groups that make use of SU-8 to construct devices to be used in many areas such as biomedical. *Klejwa et al* [16] fabricated a three axis micro strain gauge for biological application. Silicon micromachining can be used to create one-axis force sensors on a planar surface in order to study cellular traction and adhesion forces. In their previous works, poly-dimethylsiloxane (PDMS) was used to fabricate arrays of micro-needle-like structure to measure biological forces in two-axis via optical measurement of needle tip displacement. The group fabricated a device which is transparent that allow visual observation and force measurement. This device is operating in three-axis mode and force sensing mechanism is by continuous synchronous data acquisition. In order to achieve transparencies, SU-8 is used. Figure 2.8 (a) shows the schematic for the sensor and Figure 2.8 (b) is the actual optical micrographs of the SU-8 sensors.

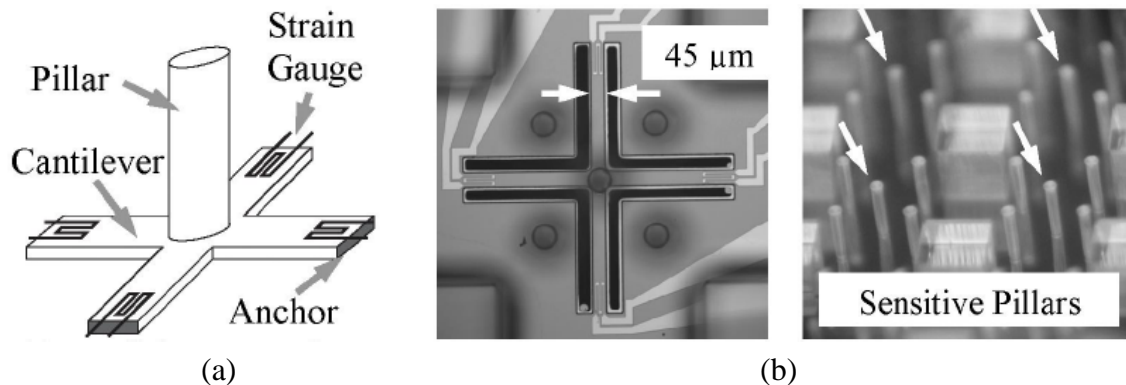


Figure 2.5: (a) Schematic of single sensor and (b) optical micrograph for fabricated sensor

2.2.4 FABRICATED SU-8 DEVICE FOR MICRO MANIPULATION

Kim et al [17] fabricated nickel microgripper with SU-8 adaptor for heterogeneous micro/nano assembly applications. The reason for having the SU-8 adaptor is that it will provide mechanical support and electrical isolation for the electroplated nickel microgripper and as well as ease of handling. The fabricated SU-8 adaptor is approximately 50 μm thick. Figure 2.6 (a) shows the schematic diagram of metallic microgripper with SU-8 adaptor and Figure 2.6 (b) is the optical micrograph image of the microgripper manually picked-up at the SU-8 adaptor notch by tweezers.

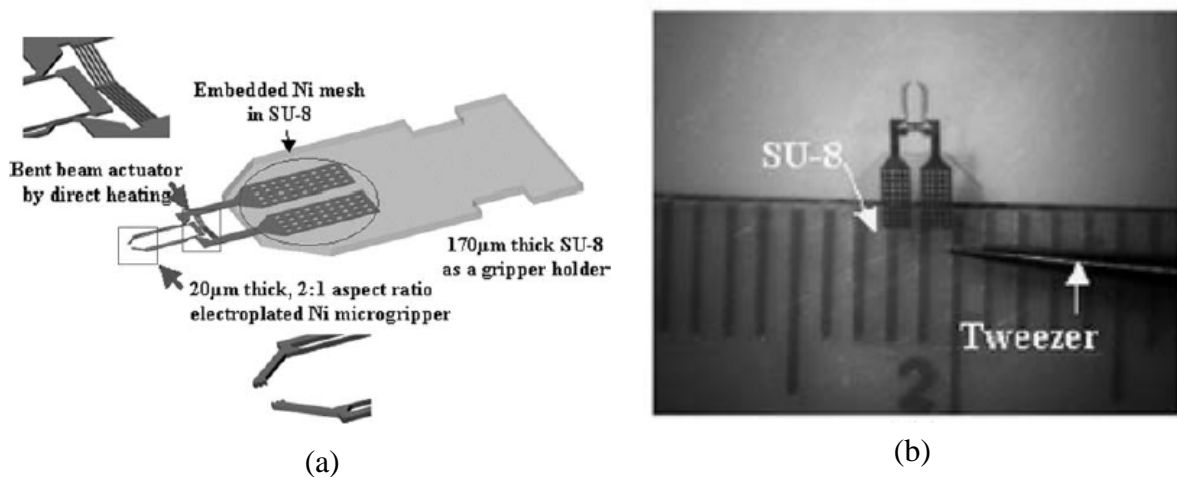


Figure 2.6: (a) Schematic diagram of microgripper with SU-8 adaptor and (b) fabricated device

Chronis et al [18] fabricated the entire gripper device with SU-8. From the paper published by the group, SU-8 has good coefficient of thermal expansion (CTE), relatively large elastic modulus and higher glass transition temperature (above 200°C). With those properties, rigid mechanical structures can be constructed for various applications. Therefore with high CTE value and high aspect ratio characteristics of SU-8, microgripper can be fabricated and actuated electrothermally.

The SU-8 thickness of device is 20 μm . Figure 2.7 shows the scanning electron micrographs of the actual fabricated SU-8 microgripper.

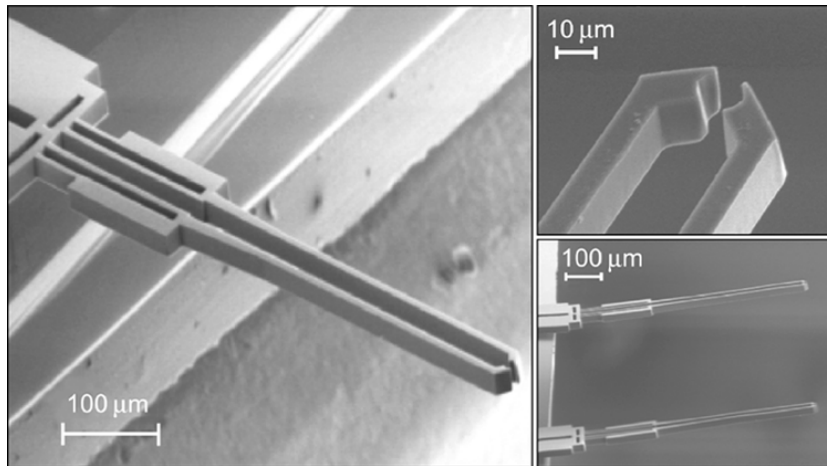


Figure 2.7: Scanning electron micrographs of fabricated SU-8 microgripper

2.2.5 FABRICATED SU-8 DEVICE FOR SIGNAL TRANSMISSION APPLICATION

Waveguide devices can be fabricated using SU-8. From the paper published by *Nordström et al* [19], it shows the capability for SU-8 to be used for light transmission application in biochemical detection. Theoretical simulations were performed in order to study the output waveguides profile and conclude the performance of the fabricated device. The group has generated square core design with height of 4.5 μm which makes the geometrical contribution to birefringence negligible. SU-8 is an isotropic cross-linked material with ladderlike structure, therefore contribution is redundant. In order to produce flexible waveguides, SU-8 is added with mr-L XP. Figure 2.8 show the scanning electron micrographs of the single mode SU-8 waveguide.

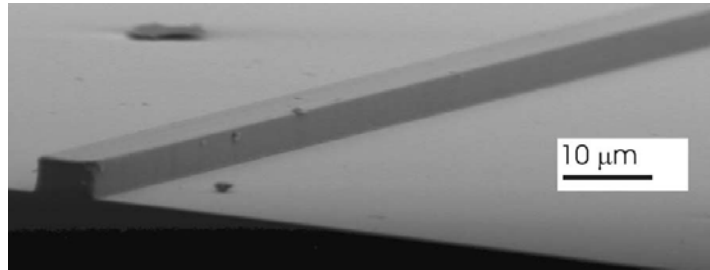


Figure 2.8: Scanning electron micrographs of fabricated SU-8 waveguide

Four types of tests were carried out. They were refractive index measurement, film stress measurement, cut-back measurement and mode profile analysis. The refractive index measurement showed that the results are highly dependent on both exposure time and temperature at which it is cross-linked. When the temperature increases from 60 °C to 110 °C, the refractive index reduces. If exposure dosage increases, refractive index also reduces. Exposure dosage doesn't seem to affect the refractive index at lower temperature. The stress measurement of the film clearly shows that the value of refractive index is inversely related to the stress for SU-8 and mr-L XP. SU-8 has slightly higher stress optical coefficient as compared to mr-L XP which has slightly lower value. Investigation of absorption of water into the polymer matrix was also carried out. The reduction in the refractive index could have been caused by the residuals of solvent in the polymer.

The authors concluded that a single-mode waveguides can be fabricated using monolithically polymeric material SU-8. SU-8 is also suitable for Micro-Optical Electro-Mechanical System (MOEMS) applications. They have studied the effects on refractive index and shown that waveguides of this type can be easily fabricated with

SU-8 by UV lithography which allows for fast fabrication of complex lab-on-chip with integrated optics.

2.2.6 FABRICATED SU-8 DEVICE FOR BIOLOGICAL ANALYSIS

Besides using SU-8 to construct microgripper, waveguide etc, some groups used it to fabricate fluidic channel for microfluidic application. *Moreno et al* [20] fabricated a simple and low cost SU-8 pressurized microchamber for pressure driven microfluidic applications. The group proposed design to achieve a fixed and controlled pressure sealing operation. The whole system consists of inlet port, control microchannel and chamber to store pneumatic energy. Figure 2.9 shows the physical schematic design of the device.

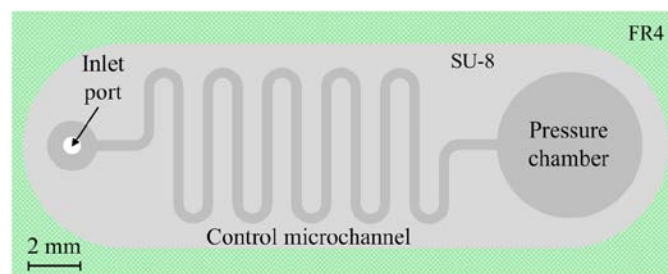


Figure 2.9: Schematic of the device design

Figure 2.10 (a) and Figure 2.10 (b) show the fabricated device before and after pressurization step. The total dimensions of the device are approximately 10x25x1.6 mm³ with a microchamber internal volume of 4 μ L and with a width of the control microchannel of 400 μ m. When operating at high pressure values, the chamber diameter must be reduced in order to reduce the mechanical stress induced in the SU-8 structure.

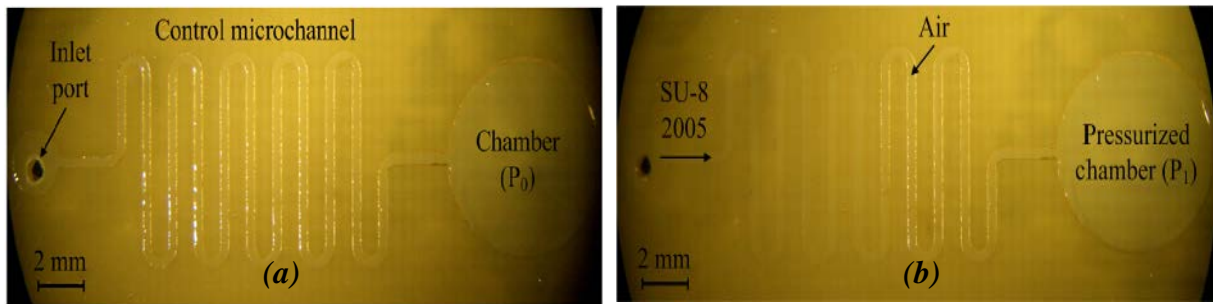


Figure 2.10: Fabricated device before chamber pressurization (a) and after chamber pressurization with crosslinked SU-8 fills part of the channel (b)

The authors concluded that the main advantages of this work lies on the time-effective fabrication, its simplicity, robustness and low cost. With SU-8 as the structural material, the device can store pressurized air for fluid impulsion without losing its pressure after a few days. As a result, it can be portable and avoid use of external macro-scale pumps and can be successfully incorporated to the market of portable microfluidics.

2.3 SACRIFICIAL LAYER METHOD FOR LIFTING OFF SU-8 FILM

SU-8 has been commonly used for high-aspect ratio structure fabrication. As mentioned in the previous chapter, it has been used for biological application as Polymerase chain reaction (PCR) analysis which requires micro-fluidic channel fabrication. Normally, SU-8 has been used as a casting mould for Polydimethylsiloxane (PDMS) imprinting. However, SU-8 has also been used to produce stand-alone lab-on-chip devices. In order to obtain the whole device after fabrication, special technique of releasing the fabricated device needs to be used. The technique used is the lift-off method. By making use of a layer of material as sacrificial layer, the whole process can easily be achieved. This section surveys a number of researches conducted by different groups on using sacrificial layer for lift-off process of SU-8 film.

2.3.1 USING POLYDIMETHYLGLUTARIMIDE (PMGI)

Polydimethylglutarimide (PMGI) is a deep UV positive resist used for bilayer lift-off process. SU-8 based microfluidics uses lift-off-resist (LOR) formulated from PMGI content as an unpatterned lift-off layer and also as a sacrificial layer for fabricating SU-8 based cantilevers. PMGI-SF series resist has lower solubility than LOR which allows higher selectivity during photo-patterning process. PMGI-SF resist is a good candidate as sacrificial layer as it is spinable with a wide range of thickness available and having photo-patternable with glass transition temperature of

190 °C which is higher than SU-8. Multiple layer micromachining processes are used in producing SU-8 structures for both mechanical and microfluidic devices.

Foulds et al [21] used PMGI as sacrificial layer for SU-8 process. Their work consists of 3 different types of processes. The mentioned advantages of using PMGI material are the ability to photo-pattern the sacrificial layer and the ability to perform post development exposure and hard baking on SU-8 layer.

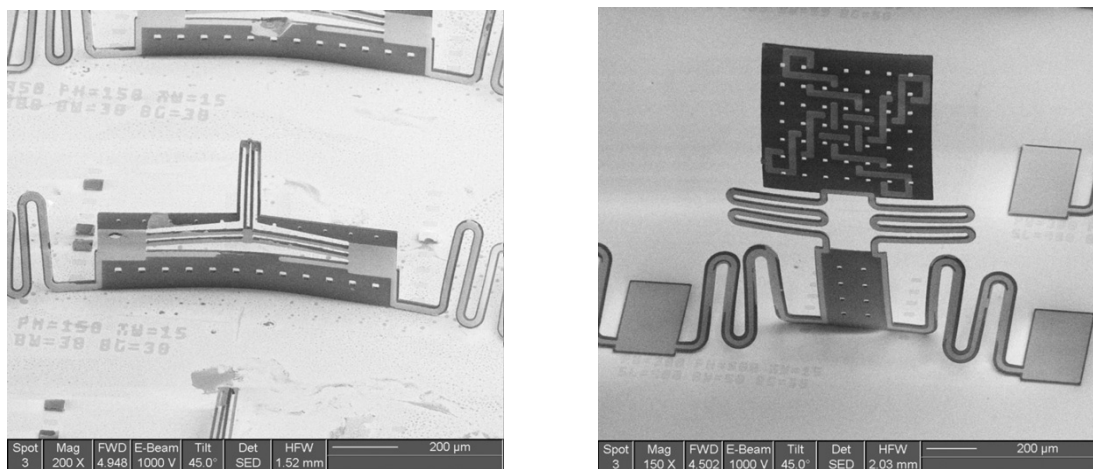


Figure 2.11: Lift-off SU-8 gripper with out-off plane movement

In conclusion, this group developed a process called polymer-on-PMGI or POP which consists of single structure with patterned metal layer. This brings advantages such as low equipment requirements with shorter duration on processing.

2.3.2 TWO SACRIFICIAL LAYER TECHNIQUES

Schmid et al [22] presented a technique of fabricating free standing polymer micro structures by applying two sacrificial layers. The sacrificial layer must be removed with the etchant easily and should not attack on the actual polymer structure material. Besides that, it must be possible to deposit and pattern SU-8 films with a thickness of 1 μm . The sacrificial layer must be able to withstand the processing temperature of high T_g of the polymer material coated on top of it. This temperature could range between 100 $^{\circ}\text{C}$ to 180 $^{\circ}\text{C}$ but can be as high as 400 $^{\circ}\text{C}$ for polyimide material. Sacrificial layer should not cross-mix with the actual polymer layer coated above it. In addition, for electrostatically actuated polymer micro structure, it must be compatible with electrodes provided by the substrate. Hence, the group selects copper and lift off resist (LOR) for their experiment testing. Figure 2.12 shows the SEM images obtained from SU-8 fabricated cantilever with copper sacrificial layer technique (a) and LOR sacrificial layer technique (b).

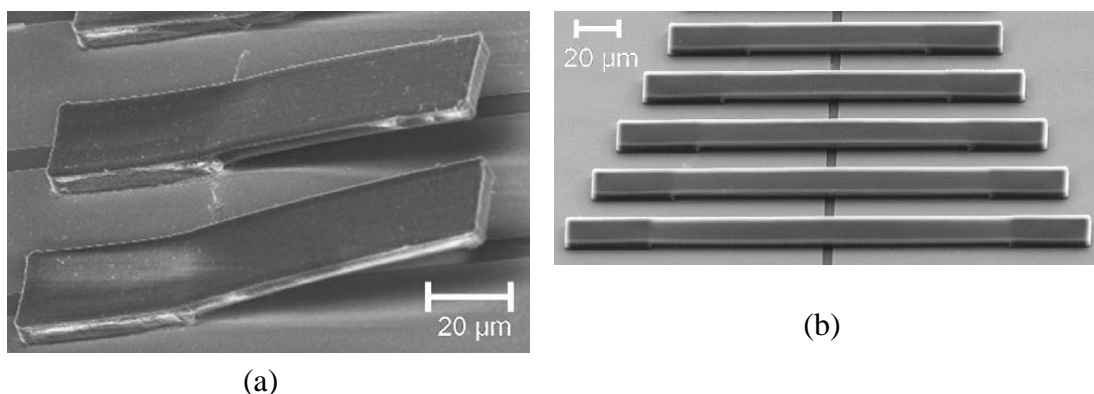


Figure 2.12: (a) SU-8 cantilever with copper as sacrificial layer technique (b) LOR as sacrificial layer technique

The author concluded that Cu and LOR can be used as sacrificial layer material for fabricating freestanding polymer micro structures.

2.3.3 USING UNCROSSLINKED SU-8 AS SACRIFICIAL LAYER

Chung and Allen et al [23] findings on sacrificial layer show that using copper as sacrificial layer may link to some issues. When the deposited thickness is hundred of micrometers, the selective deposition and removal of the copper layer will require additional time. Furthermore, copper is selectively removed with strong basic or acidic etchant for sufficient etch rates. And, electrodeposited copper requires additional fabrication complexity.

The group suggested that using another alternative sacrificial material which is uncrosslinked SU-8 could eliminate the above issue. As mention, uncrosslinked SU-8 have a number of properties. When the temperature is at 65 °C, SU-8 is highly chemically resistant and it can maintain a flat surface for lithography and uncrosslinked SU-8 could be easily removed. Deposition of seed layer, insulating layer or electroplating mould could be also avoided by using this method. Figure 2.13 shows the SEM images of the electrodes fabricated by using uncrosslinked SU-8 as the sacrificial layer, (a) close-up of free-standing SU-8 layer of the electrode (b) overview of the electrode where underneath SU-8 have been removed.

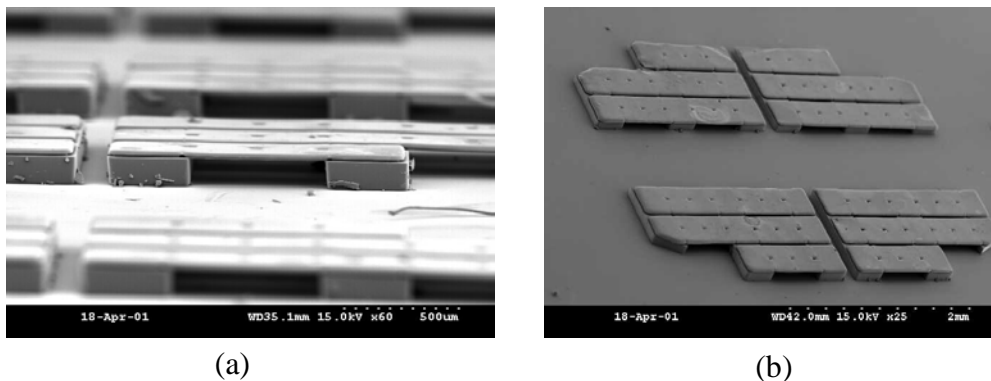


Figure 2.13: Overview of the fabricated SU-8 electrode using uncrosslinked SU-8

2.3.4 USING OMNICOAT™ AS SACRIFICIAL LAYER

Bohl *et al* [24] reported in their research publication about Omnicoat™ layer as sacrificial layer. As mention in their paper, SU-8 has limitations in constructing multilayer structure due to the fact that SU-8 is a negative resist.

In order to release large SU-8 structures, the group designed a novel lift-off technique based on Omnicoat™ as it can develop selectively against SU-8. However, it is not effective in removing large functional structures. Omnicoat™ layer with thickness of less than 100 nm provides very small gaps for the developer to pass through and etch off the SU-8 film. One solution to overcome this issue is coating thicker layer of Omnicoat™. Thicker the Omnicoat™ layer, the lower the adhesion between SU-8 film and silicon surface. If the adhesion is weak enough, stress in the SU-8 can cause the SU-8 film to peel off pre-maturely. The cross-linking process within the SU-8 during curing causes such stress to form at the silicon-SU-8 interface due to the effect of volume shrinkage of the SU-8 layer. The stress induced at the material interface increases with the lateral dimensions and the height of the SU-8 structures. Caused by the lowered adhesion, the SU-8 structures are released from the substrate during development if the right layer of Omnicoat™ is not selected. In order to speed up the entire process, ultrasonic bath can be used. Figure 2.14 (a) shows the photograph of the SU-8 device after the lift-off process and Figure 2.14 (b) shows the SEM image of the SU-8 device fabricated by SU-8 lift-off technology.

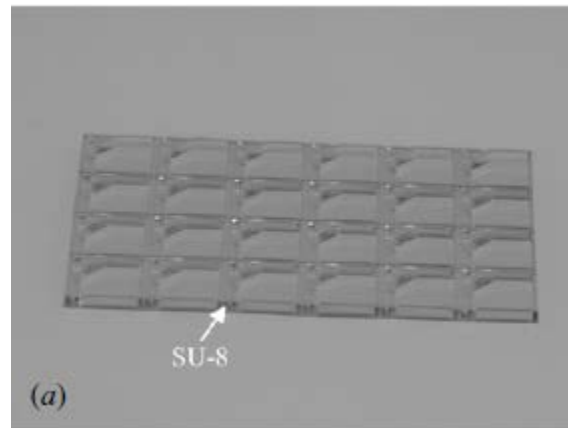


Figure 2.14 (a) Photograph of a Dispensing Well Plate (DWPTM) after lift-off with lateral dimensions of 27 mm \times 18 mm and a height of about 551 μ m.

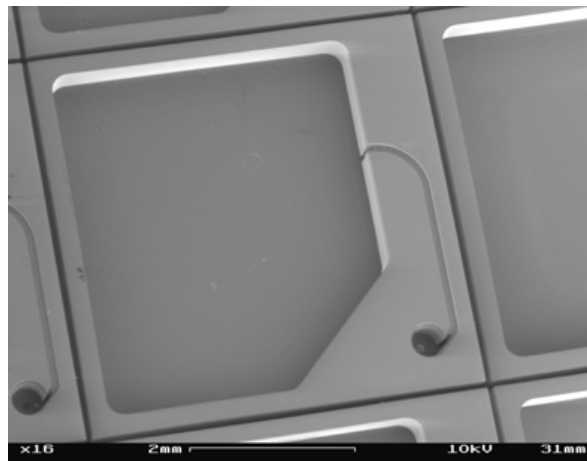


Figure 2.14 (b) SEM image of a Dispensing Well Plate (DWPTM) using SU-8 lift-off technology

2.3.5 USING AZ 9620 PHOTORESIST AS SACRIFICIAL LAYER

J. Zhang et al [25] reported on using AZ 9620 positive photoresist as the sacrificial material for constructing SU-8 polymer structure. In order to construct SU-8 structures, two or more steps of photolithography process are needed. The whole fabrication process consists of:

1. First layer of SU-8 coating and patterning.
2. Without developing step, thin film such as metal films, parylene films, etc are deposited on the SU-8 surface and used as insulation layer.
3. Insulation layer are patterned.
4. Second layer of SU-8 layer are spin-coated and patterned
5. Wafers are dipped into developer for developing process with agitation ultrasonically.

Figure 2.15 shows the embedded microchannel using AZ 9620 as sacrificial layer.

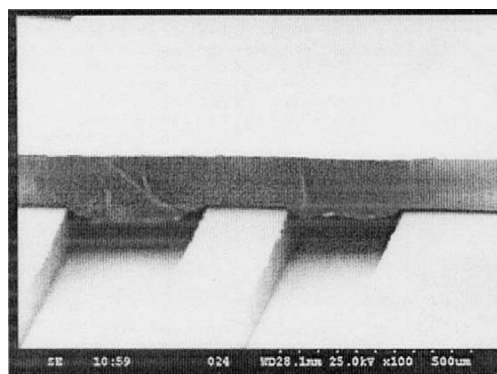


Figure 2.15: Microchannel using AZ 9620 as sacrificial layer

CHAPTER 3 – THEORY AND WORKING PRINCIPLE

3.1 STRUCTURE AND PHYSICAL PROPERTIES OF POLYMERS

3.1.1 PHYSICAL STATES OF POLYMERS

Polymers are present in four physical states, crystalline and three amorphous states (glassy, rubbery and viscous flow). The solid polymers which are glassy or crystalline are named as rigid polymers. Every specific state has its own complex mechanical properties and has its own unique technical applications.

In order to determine the degree of compliance of polymer, thermo-mechanical characterization can be done. At temperature range lower than glass transition temperature T_g , polymers deform in the way of glass. Significant increase in reversible strain occurs at temperatures above T_g , indicating the rubbery state.

3.2 MECHANICAL PROPERTIES OF POLYMER

3.2.1 PROCESSING CONDITIONS AFFECTING THERMAL AND MECHANICAL PROPERTIES OF SU-8

Thermal and mechanical properties will be affected by the influences of curing conditions such as baking temperature which is inclusive of pre-baking, post-exposure baking and hard-baking, baking duration and UV dosage. This can be shown by the results published by *Feng et al* [26].

From the conclusion, glass-transition temperature (T_g) of SU-8 is the same for the baking temperature range between 25 °C to 220 °C and a duration of 20 minutes. The limiting glass-transition temperature (T_g) will be approximately 240 °C once the cross-linking reaction have completed. Heat shrinkage will occur at the peak temperature obtained from the temperature plot and with a factor of 1.16 times higher than the baking temperature range. Both glass-transition temperature (T_g) and shrinkage temperature will definitely be affected by baking duration.

Table 3.1: Mechanical properties of SU-8 before and after different process conditions

Sample	PEB duration (mins)	HB duration (mins)	Tested immediately after processing			Tested 24 h later after processing		
			Modulus (GPa)	Strength (MPa)	Elongation (%)	Modulus (GPa)	Strength (MPa)	Elongation (%)
1	0	0	0.7	16.1	24.0	0.7	14.9	7.5
2	3	0	-	-	-	1.6	35.9	3.5
3	5	0	1.7	37.2	3.9	1.9	33.4	2.6
4	15	0	2.2	48.3	5.9	2.5	44.8	3.0
5	30	0	2.4	52.6	3.8	2.6	52.5	2.7
6	30	1	-	-	-	2.5	44.6	2.0
7	30	5	2.4	73.1	5.2	-	-	-
8	30	30	-	-	-	2.7	42.1	1.8

According to the results presented in Table 3.1, sample before post-exposure bake (PEB) are more ductile with higher elongation of 30%. However, elongation value dropped to 7.5% after being exposed to ambient environment for duration of 24 hours which is caused by evaporation of the solvent.

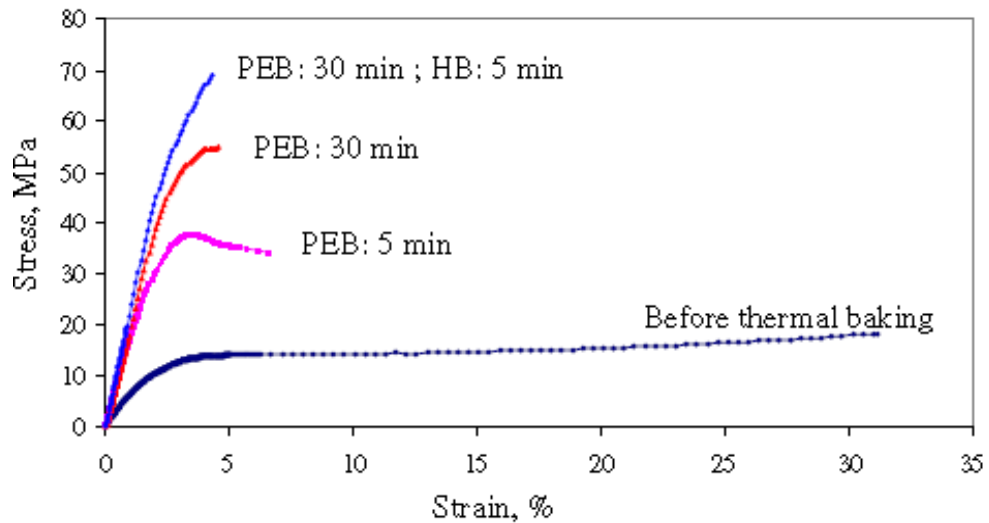


Figure 3.1: Stress-strain curves for SU-8 at before and after post-exposure bake duration with other conditions [26]

From the stress-strain curves (Figure 3.1), it is shown that the stress in SU-8 will increase with respect to the post-exposure baking (PEB) duration. For instant, PEB of 5 minutes have stress value of approximately 35 MPa. When PEB duration increases to 30 minutes, stress value increases to approximately 65 MPa. However when PEB duration remained at 30 minutes followed by 5 minutes of hard baking, the stress increases to approximately 70MPa.

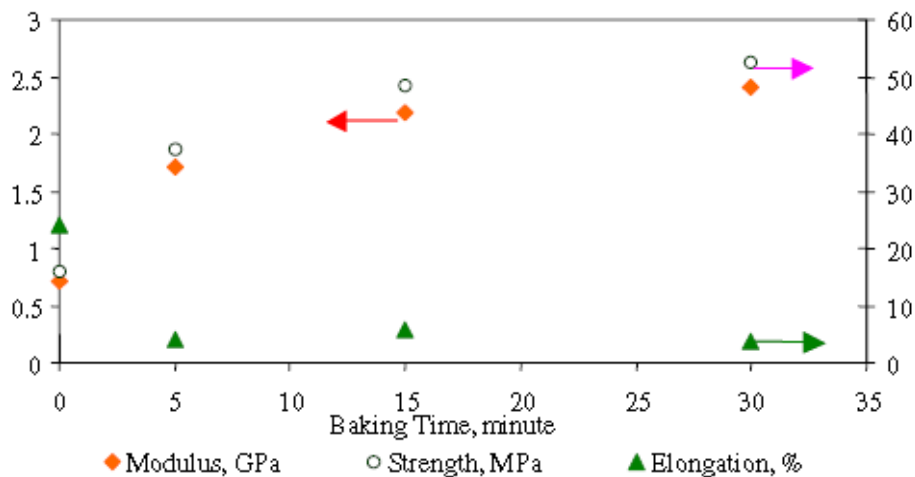


Figure 3.2: Change in tensile properties with respect to baking time [26]

Result of Figure 3.2 shows the effect of post-exposure bake (PEB) on the mechanical properties of SU-8. The cross-linking reactions are only active when the baking temperature is higher than the glass transition temperature of the material. Reaction will slow down and effectively halt when T_g of SU-8 reaches close to baking temperature.

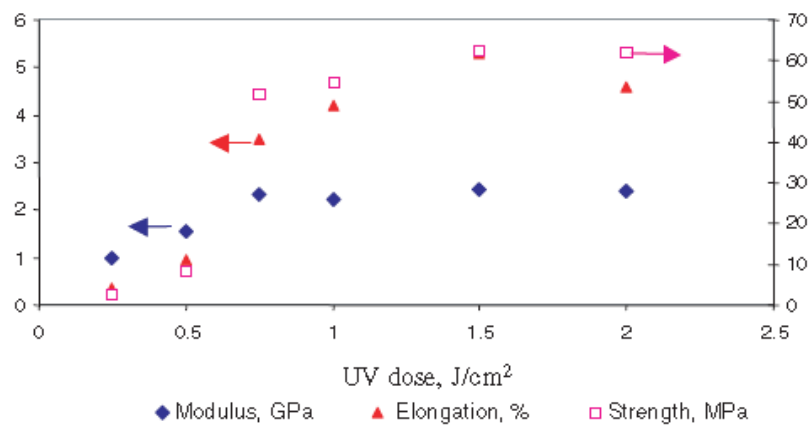


Figure 3.3: Change in mechanical properties with respect to effect of UV dosage [26]

Another factor that will influence the tensile properties of the resultant SU-8 coating is the ultra-violet (UV) exposure dosage which will be applied during the exposure step. Figure 3.3 shows the influence of UV dose on the properties of the films after baking at 95 °C for 30 minutes. The integrity of SU-8 film improved dramatically when the UV dose is below 1 J cm⁻² and reaches plateau after this dosage level. The reason for such phenomenon is because of the photo-acid generator present in the resist system that absorbs photons and produces a strong acid when exposed to UV source. This particular type of strong acid acts as the catalyst for cross-linking reaction to happen during post-exposure bake and hard baking stage. The reaction rate of cross-linking to take place will depend on the concentration of the catalyst which is decided by the amount of UV dosage.

Therefore, in conclusion, effect of UV dose on glass-transition behaviour of SU-8 will be very different for sample before and after thermal baking. Factors such as tensile and mechanical properties show changes with baking temperature.

CHAPTER 4 – MICROFABRICATION AND RELEASE OF SU-8 STRUCTURES

4.1 EQUIPMENT (SAMPLE PREPARATION)

4.1.1 SPIN COATER AND HOT PLATE

CEE 100 Brewer Science spin coater (Figure 4.1) is used to coat AZ 4620 positive photo-resist and SU-8 film onto 4 inch silicon wafer.



Figure 4.1: Brewer Science CEE 100 spin coater for coating film onto substrate

Once the coating process is completed, baking process can be carried out on SAWATEC, HP-150 hotplate (Figure 4.2). It can be used for standard soft bake and hard bake processes in lithography application. The temperature range is designed up to 250 °C. And it also offers high uniformity and process repeatability capabilities.



Figure 4.2: SAWATEC HP-150 hotplate for baking process

4.1.2 MASK ALIGNER

SUSS MicroTec MA/BA 8 mask aligner (Figure 4.3) is used for lithography application. Thick resist performance can also be done with this system. It can provide a UV wavelength range of 365 nm and 405 nm which is suitable for SU-8 film cross-linking application. It can achieve the minimum feature size of 1 μm . Substrate size can be in irregular to standard 8 inch in size. Mask size allowable typically ranging from 3", 5", 7" and 9". There are a few exposure modes such as soft, hard, vacuum and proximity contact.



Figure 4.3: SUSS MicroTec MA/BA 8 Mask aligner for patterning transfer

4.1.3 WET BENCHES

Solvent wet benches (Figure 4.4) are used to develop the exposed SU-8 film where wet chemical such as SU-8 developer and Isopropyl alcohol (IPA) will be used. Pre-cleaning process such as piranha treatment will be carried out at acid wet benches.



Figure 4.4: Wet benches where developing work is carried out

4.1.4 DIP COATING SYSTEM

In-house built dip coater (Figure 4.5) is used to coat other film such as PFPE onto SU-8 film before tribological testing with dipping and withdrawal speeds of 1.9 mm/s



Figure 4.5: Dip coating system

4.1.5 OXYGEN PLASMA TREATMENT SYSTEM

Harrick Plasma (PDC-32G) (Figure 4.6) is a system which is used to generate oxygen plasma and provide plasma bombardment on the silicon substrate surface before overcoating with AZ 4620 thick positive photoresist. The maximum radio frequency (RF) power deployed by this system is 18 W and we deployed this power for our sample use.



Figure 4.6: Harrick Plasma (PDC-32G) used for the oxygen plasma treatment on AZ 4620 positive photo-resist sacrificial layer

Oxygen plasma can remove organic contaminants by chemical reaction with highly reactive oxygen radicals and through ablation by energetic oxygen ions. It can also promote surface oxidation and hydroxylation (OH groups); increasing surface wettability. Oxidation may be undesirable for some materials (e.g. gold) and can affect surface properties.

4.2 EQUIPMENT (TESTING AND MEASUREMENT)

4.2.1 TRIBOLOGICAL TESTER

CETR UMT-2 microtribometer (Figure 4.7) is used to perform friction and wear tests and the main result obtained from the system is the coefficient of friction (COF) with respect to time which later needs to be converted to number of cycles. A Si_3N_4 ball of 4 mm diameter (with a roughness of 5 nm) was used as the counterface.

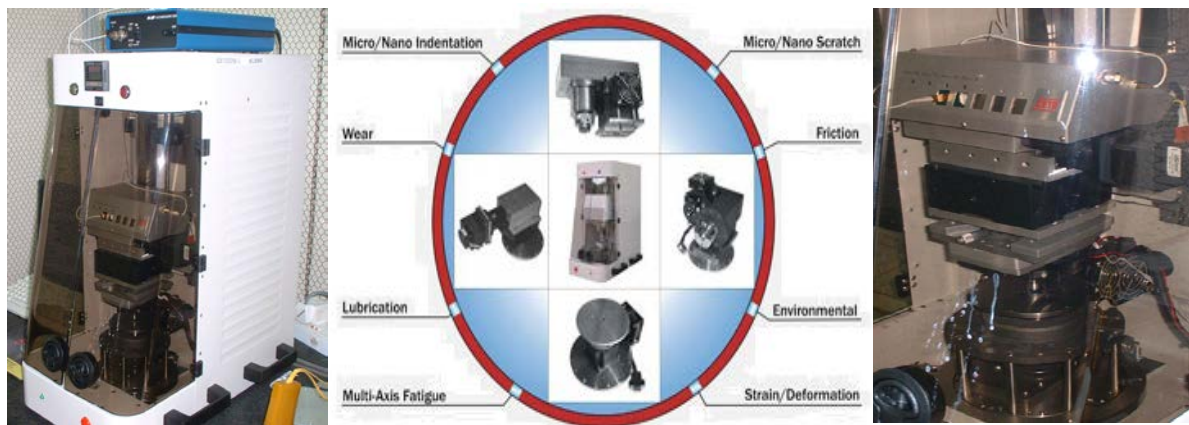


Figure 4.7: CETR UMT-2 micro-tribometer to perform tribological testing

4.2.2 GONIOMETER (CONTACT ANGLE MEASUREMENT)

Contact angle and surface free energy of different specimens were determined by VCA Optima Contact angle System (AST product, Inc., USA) as shown in Figure 4.8. By conducting contact angle measurements, apparent surface free energy could be determined and the liquid used for this measurement was distilled water. The droplets size of distilled water used in the measurements was 0.25 μl .



Figure 4.8: VCA Optima Contact angle System use for water contact angle and surface energy analysis

4.2.3 FAILURE ANALYSIS EQUIPMENTS

Various failure analysis equipment such as Olympus optical microscope (BX60) (Figure 4.9), KLA-Tencor surface profiler (P-10), Hitachi field emission scanning electron microscope (FESEM) (S-4300) are used to study the lifted SU-8 film structure.




Figure 4.9: Various failure analysis equipment such as microscope, contact profiler and SEM respectively

4.3 SUMMARY OF EXPERIMENTAL SEQUENCE

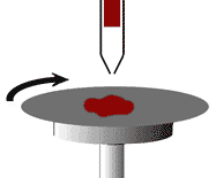
In order to obtain the release SU-8 structures, a piece of silicon wafer with size of 4 inches was used. First process step is to clean the wafer with chemical consist of the mixture of sulfuric acid and hydrogen peroxide. The solution is mixed and boiled to the required temperature. The cleaning process duration took about 5 minutes to complete. After drying of wafer with nitrogen gas, positive photoresist was spread onto the surface of the wafer and spanned at the require speed. Next, the coated layer was dried by hot plate and the coating process repeated again till it reaches the required thickness. SU-8 epoxy was dispensed on the wafer overcoat with resist and soft baking of SU-8 was carried out in order to dry away solvent contain in within the polymer. Patterning was done using a photomask with required UV dosage and duration. After UV exposure was completed, the sample will go through post exposure baking on the hotplate. Finally, the baked sample will be developed using SU-8 developer.

Table 4.1 summarizes all sequential processes being carried out until the lifted SU-8 structure is obtained.

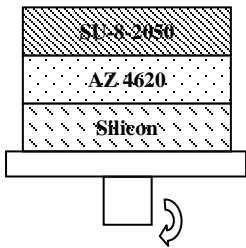
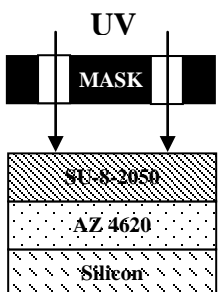
Table 4.1: Basic process steps

Step No:	Process Description	Parameters	Results
1	Use of silicon wafer as fabrication substrate	<ul style="list-style-type: none"> • Size: 4”(100 mm) • Thickness: 525 μm • <100> • P type • Single side polished 	
2	Using chemicals to remove any contaminants from the wafer	<ul style="list-style-type: none"> • 7:3 of H_2SO_4, H_2O_2. • Applying 90 $^{\circ}\text{C}$ to 120 $^{\circ}\text{C}$ to boil the solution. 	<ul style="list-style-type: none"> • Surface will have higher wettability. • Cleaning of organic

MICROFABRICATION AND RELEASE OF SU-8 STRUCTURES

		<ul style="list-style-type: none"> • Rinse the sample with DI water for 5 minutes. • Dried sample using N₂. • Dehydration of substrate at 100 °C for 2 minutes. 	contaminants
3	Coat Si wafer with AZ 4620 positive photo-resist	<ul style="list-style-type: none"> • Spin coater speed is 4000 RPM • Duration is 1min 	 <ul style="list-style-type: none"> • Single coat of AZ 4620 is about 5 to 8 μm
4	Dry AZ 4620 sacrificial layer	<ul style="list-style-type: none"> • Set temperature of hotplate to 110 °C to 120 °C stated for AZ 4620 manufacturer specification (<i>Appendix B</i>) 	

MICROFABRICATION AND RELEASE OF SU-8 STRUCTURES

5	Repeat of coating for AZ 4620 positive photo-resist and soft baking		<ul style="list-style-type: none"> 5 rounds of coating and baking in order to obtain the expected thickness
6	Coat AZ 4620 positive photo-resist layer with SU-8-2050	<ul style="list-style-type: none"> Spin coater speed is 500 RPM Duration is 1min 	 <ul style="list-style-type: none"> Expected thickness 200 μm
7	Soft baking of SU-8 layer on hotplate	<ul style="list-style-type: none"> Set soft baking temperature of hotplate to 65 °C for 7 minutes. Rise the baking temperature of hotplate to 95 °C for approximately 30 to 45 minutes. 	<ul style="list-style-type: none"> As thickness increases, soft baking duration require to lengthen in order to drive away the solvent trap in SU-8 film
9	Patterning using mask aligner	<ul style="list-style-type: none"> Expose the soft cured SU-8 film with i-line ultra-violet rays at wavelength of 365 nm for duration of 15 to 30 second. 	
10	Post exposure bake	<ul style="list-style-type: none"> Set baking temperature of hotplate to 65 °C for 1 minute Rise the baking temperature of hotplate to 95 °C for approximately 5 minutes 	<ul style="list-style-type: none"> Exposed area will be cross-linked and harden. However unexposed area will remain as it original state.

MICROFABRICATION AND RELEASE OF SU-8 STRUCTURES

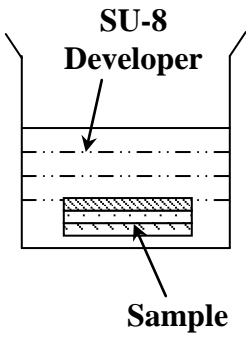
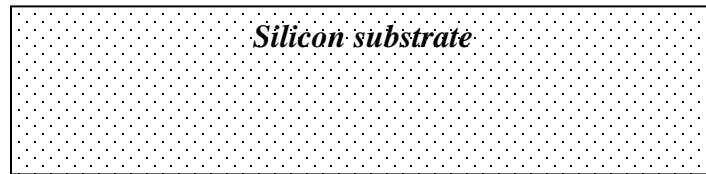
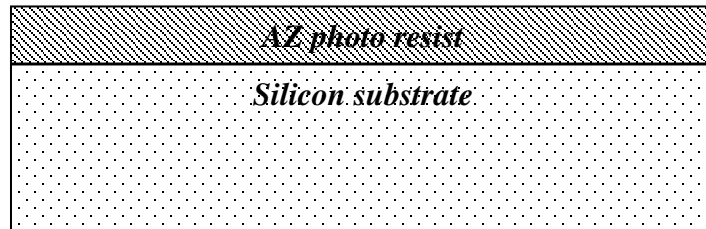
11	Developing of exposed SU-8 film in developer	<ul style="list-style-type: none"> Developing of the harden structure can be done by soaking the wafer in a beaker filled with organic solvent (propylene glycol monomethyl ether acetate) Lifting off of the whole structure normally take 20 to 30 minutes, depending on the thickness and area of the film Once after lift-off process completed, do not use DI water to rinse as DI water may produce residue. However use isopropyl alcohol (IPA). Dried sample using N₂. 	
12	Hard bake	<ul style="list-style-type: none"> Rise the baking temperature of oven to 150 °C for approximately 15 minutes 	

Figure 4.10 show the complete process flow of the fabrication and release process. It comprises of coating process, pattern transfer stage, developing process which include the releasing of the SU-8 film layer. From the cross-sectional view presented, it shows the whole process of releasing the developed structures starting from positive resist coating as sacrificial layer, overcoating of SU-8 layer, patterning of the SU-8 structure and developing of the final released structure.

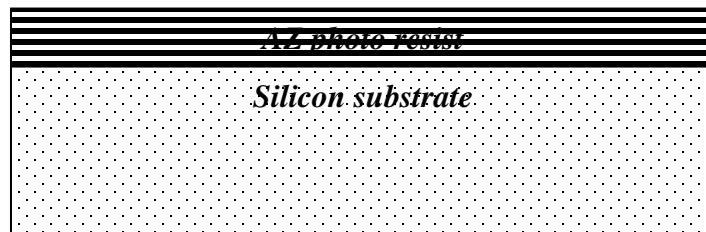
MICROFABRICATION AND RELEASE OF SU-8 STRUCTURES



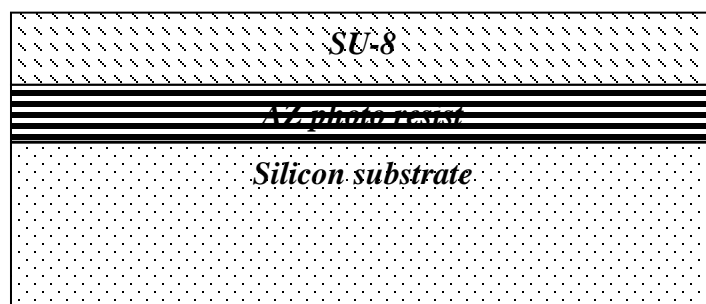
Step 1: Preparing clean surface of silicon for fabrication procedure



Step 2: Spin coating of AZ photo-resist on silicon substrate and soft baking



Step 3: Multiple stacking of AZ photo-resist using step 2



Step 4: Spin coating of SU-8 on top of AZ layer and applied pre-exposure baking

Figure 4.10: Process flow of SU-8 fabrication and releasing process, Step 1 – Step 4

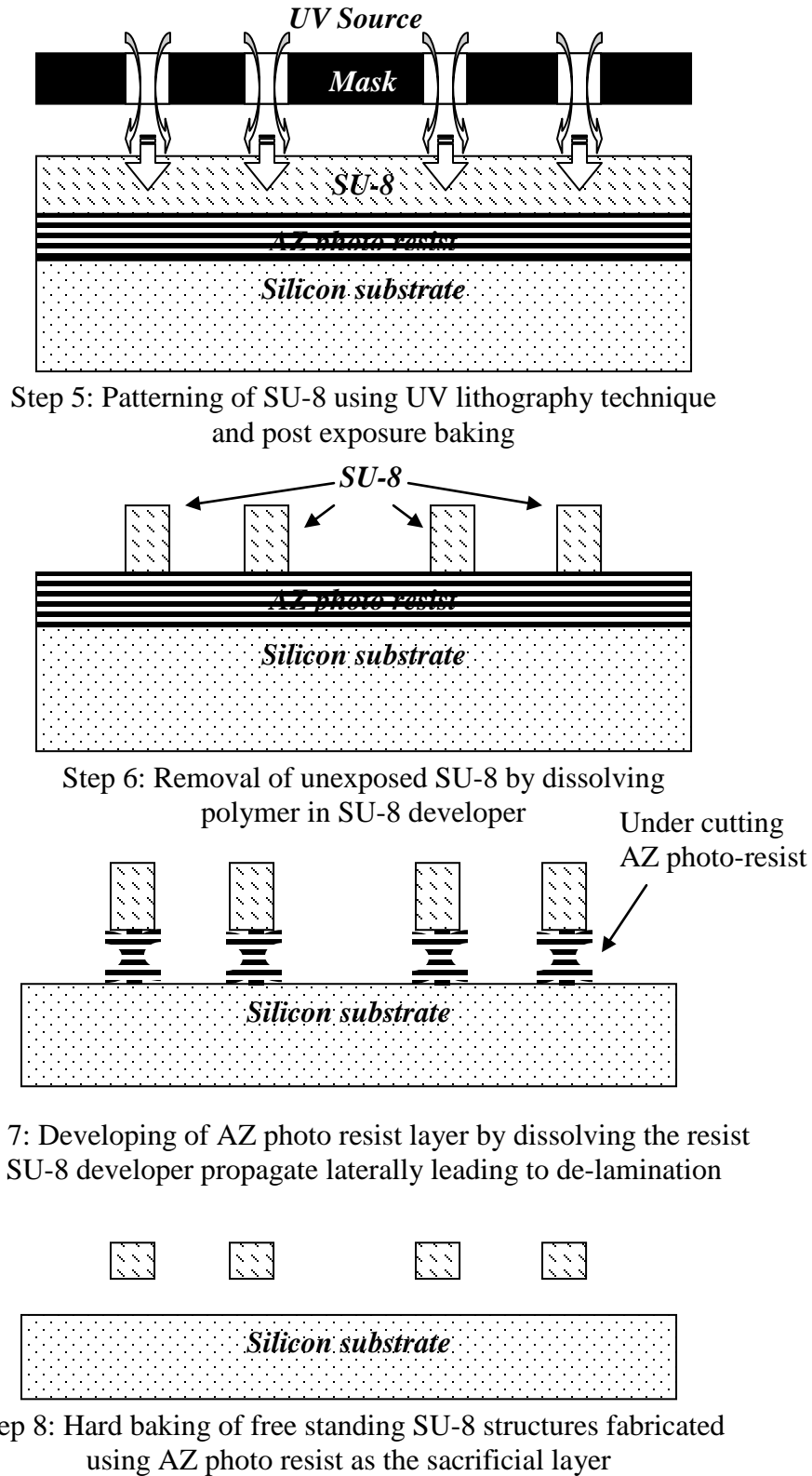


Figure 4.10: Process flow of SU-8 fabrication and releasing process, Step 5 – Step 8

4.4 PHOTOLITHOGRAPHY PROCESSES

In order to produce samples for testing, dark-field photomask was used. SU-8 is UV sensitive and it cross-links when UV light is shone on it. On the other hand, those areas that are not exposed to UV are not cross-linked and hence can be dissolved in the developing solvent. Therefore in order to achieve it, we must apply a dark-field photomask. In order to reduce the fabrication cost, we make use of transparency printed photomask. The diameter of the designed sample is between 100 micrometers to approximately 30 millimeters. However if the diameter of the sample produced is between 5 to 10 micrometers, the photomask used must be made of glass as the image transferring result will be better. Typically, photomasks are made on soda lime glass, quartz (fused silica) or polyester Film. Table 4.2 shows some characteristics between plastic transparency mask, soda lime glass mask and quartz mask.

<i>Plastic film</i>	<i>Soda lime glass</i>	<i>Quartz</i>
Low price	Good price/quality ratio	Expensive
Low resolution	High resolution	High resolution
Weak stability	Easy to clean	Very stable
Easy to handle	Stable	Can break
Wavelength > 350 nm	Can break	Wavelength < 350 nm
	Wavelength > 350 nm	

Table 4.2: Characteristics between plastic transparency mask, soda lime glass mask and quartz mask

The fabrication of a photomask requires several steps. In this section we will describe each step required for photomask fabrication. The pattern information is created using a drawing package, often in AutoCAD or other suitable software packages such as L-Edit. This data is processed into internal CAD format (Gerber) and transferred to a lithography tool which is referred as photomask writer – which then exposes the design onto the photomask substrate. Photomask writer can process for both glass and film photomasks. Once the manufacturing process is finished, the mask is cleaned and inspected. Figure 4.11 (a) shows the transparency photomask pattern used to produce gears which are of different sizes. Figure 4.11 (b) shows the transparency photomask pattern used for fabricating patterns for testing purposes such as tribological testing, surface energy analysis or surface roughness measurement and etc.

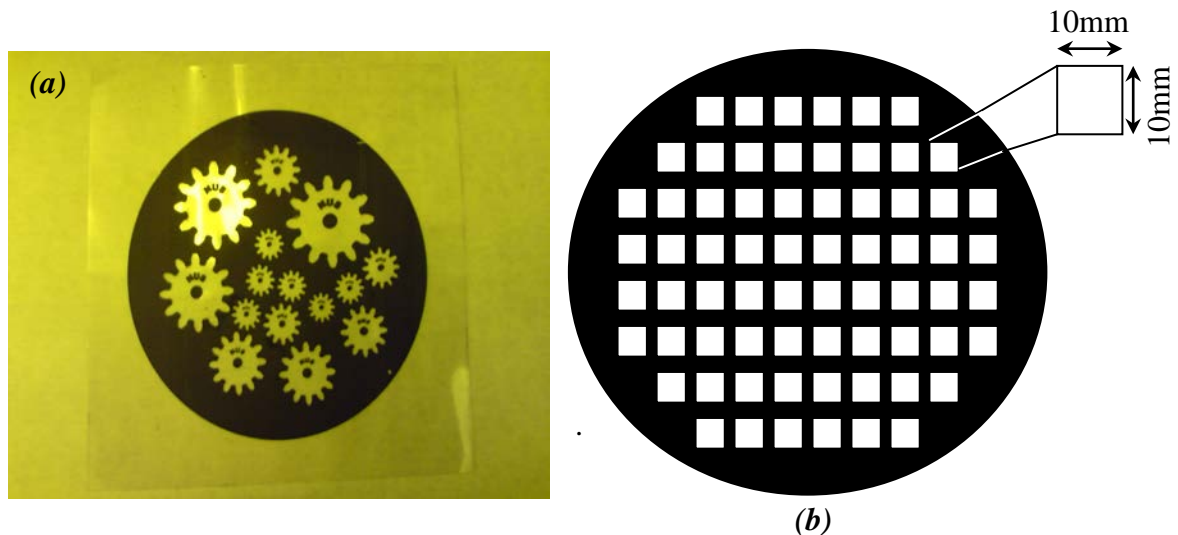


Figure 4.11: (a) Photo-image of the transparency photomask used to fabricate gears and (b) Photo-image of the transparency photomask used 10mm by 10mm test sample

CHAPTER 5 – RESULTS AND DISCUSSION

5.1 INITIAL DEVELOPMENT OF SU-8 STRUCTURES USING SACRIFICIAL LAYER TECHNIQUE

Sacrificial layer technique, also known as lift-off method, is commonly used in MEMS fabrication process. The common application of this technique is by means of metal pad construction used in electrical connection. In lift-off process a sacrificial material, such as photoresist, is first deposited and patterned on the substrate. The material of interest is then deposited on top and the sacrificial material is subsequently removed, leaving behind the structure lifted-off from the substrate. These processes are useful for patterning materials that cannot be etched without affecting underlying materials on the substrate. There are some considerations needed to be made before carrying out lift-off process. Factors include:

- Type of lift-off material used
- Material to be deposited and patterned by lift-off process
- Thickness of the deposited materials

The reason of knowing the type of lift-off material used is important so that correct developer or etchant can be used to dissolve this layer. The second factor is important for knowing if this etchant or developer will damage the device layer coated on top of the sacrificial layer. And lastly, thickness of deposited materials needs to be understood as this will affect the developing time or etching time of the sacrificial layer. If the duration of etching of the sacrificial layer is too long, the etchant or developer may cause damage to the device layer itself.

5.1.1 AZ4620 POSITIVE PHOTORESIST

The thickness of each single coat of AZ 4620 positive photo resist layer was in the range of 5 to 8 μ m. Therefore, in order to achieve the required thickness, multiple layers are needed. Our other objectives were to minimize the solvent content in each sacrificial layer and at the same time to achieve a shorter baking duration. Single layer coating with lower thickness consists of lesser solvent content and requires a shorter baking time to drive off and vaporize the solvent in comparison to that of a layer with higher thickness. In the case of coating thick photo resist layer using multiple coat method, the solvent content in the resist could be vaporized rapidly in the consecutive spinning and baking processes. By employing the multiple coating method to form thick sacrificial layer instead of single thick sacrificial layer, out-gassing and scission effects during longer baking duration at a higher temperature could be prevented and further, could avoid ‘popping’ effect in the fabricated SU-8 structure, much evident in the case of single sacrificial layer with higher thickness. Too much of the solvent may have some other effects on the fabrication results. For example, micro-bubbles will form and solvent will out-gas and cause the resist layer to have filled with cavities. And that will affect the flatness of SU-8 layer coated on the top surface.

5.1.2 COATING AND BAKING OF SU-8 LAYER

When dispensing the SU-8 (grade 2050) epoxy onto the substrate coated with AZ 4620 layer, an additional care needs to be taken. If the distance between the wafer surface and the dispensing unit is longer, there is a possibility of the formation of air bubbles. As the SU-8 epoxy is very viscous fluid, the trapped air bubble may lead to voids and cavities in the subsequent soft baking and they eventually weaken the structure.

During baking process, thermal stresses are introduced within the structure if additional care is not taken in setting the bake temperature and duration. A gradual increment in temperature from 65 °C to 95 °C can be used to avoid the formation of any thermal stresses which may lead to cracks and shrinkages. During soft baking, it is important that the hotplate with good thermal control and clean surface which does not contain any contaminants or particles, is used in order to achieve a better contact with the silicon substrate providing a uniform thermal distribution across the wafer and the SU-8 layer. Conventional ovens can also be used for curing but they are often not recommended because they are observed to form a skin-like layer over the SU-8 surface due to the rapid evaporation of the solvent from the surface which results in the cross-linking and curing of the monomers on the surface trapping the solvent within the structure and consequently to non-uniform curing. This type of skin-like layer formation over the surface could inhibit the vaporization of the solvent, eventually causing the structure to become soft. Therefore, a special care must be taken if the baking is carried out in an oven to avoid the above-described problem.

5.1.3 COMPARISON WITH THE EXISTING RELEASING METHODS

Various methodologies and materials which are broadly classified as metals and polymers had been deployed and investigated as sacrificial layer [26-31]. MicroChem Corp, USA has developed a material (*OmniCoatTM*) which has been used as a sacrificial layer for SU-8 coating. From the literature works, it is clear that the processing cost is very high in the case of using metal as sacrificial layers. These metallic sacrificial layers are usually formed by expensive methods such as evaporation or sputtering and the lift-off process may take 20 min to 1 hour heated etchant. However, the processing cost of the sacrificial layer coating and the lift-off time can be drastically lowered by the utilization of polymeric materials which can be easily spin-coated.

In order to obtain a good released structure, some important factors need to be considered for the selection of the right candidate for sacrificial layer. Adhesion property with respect to the SU-8 layer and the base surface material, chemical properties of dissolving medium for the sacrificial layer and cost effectiveness with respect to material and processing etc are some of the important factors which need to be considered while selecting a suitable sacrificial material. Therefore, in the current thesis work, the fabrication procedure was simplified by using AZ photo resist over other polymeric materials as sacrificial layer. The use of AZ photo resist as sacrificial layer had previously been investigated for the removal of SU-8 mould by *Dellmann et al* [8] and for the fabrication of cantilever structure by *Ezkerra et al* [37]. As per our literature survey, the AZ photo resist sacrificial layer was not

employed for lift-off procedure or releasing of SU-8 structure. However, AZ layer was sacrificed with other solvent apart from SU-8 developer during the experiments conducted by *Bao et al* [38]. Therefore, from these studies, we have selected AZ photo resist as a sacrificial layer to lift-off SU-8 structures.

The procedure for fabricating micro structures using SU-8 has been simplified in the current work to make it less expensive using easily available materials, shorter process flow and process time with high volume production-ability and repeatability with lesser resource consumption giving an edge over other procedures in cost and time, which are primary considerations in the commercialization/mass production. The procedure has been extended to fabricate hierarchical microstructure patterns with SU-8 nano-composite polymer and thereby making it a comprehensive fabrication procedure to fabricate SU-8 based microstructures.

5.1.4 RELEASE OF SU-8 MICROSTRUCTURE

The UV cross-linked area of SU-8 will be slowly developed in the developer solution with minimum amount of agitation. After 10 to 20 minutes of developing duration, the shape of the exposure pattern will start to form. If the pattern diameter is smaller, it will lift-off faster as compared to bigger size patterns. Approximately, after 30 minutes those structures with smaller diameter will start to lift-off. And once this happened, special cares are required so as not to damage the structure. It maybe rinsed in isopropyl alcohol (IPA) till the solution appears to be clear. At the beginning of the rinsing process, the alcohol solution tends to appear milky as this contains SU-8 material. If this step is not done properly, debris will form on the surface of SU-8 structure and may cause reliability issue. Once the rinsing is completed, hard-baking of the SU-8 structure maybe carried out in an oven for uniform heat distribution. Figure 5.1 shows the lift-off structure which has the thickness of 50 micrometer.

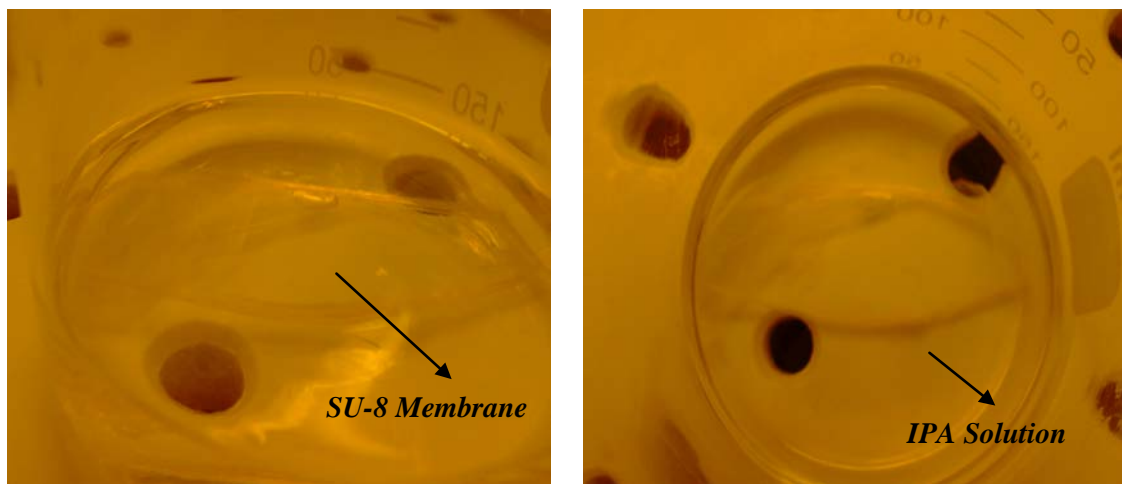
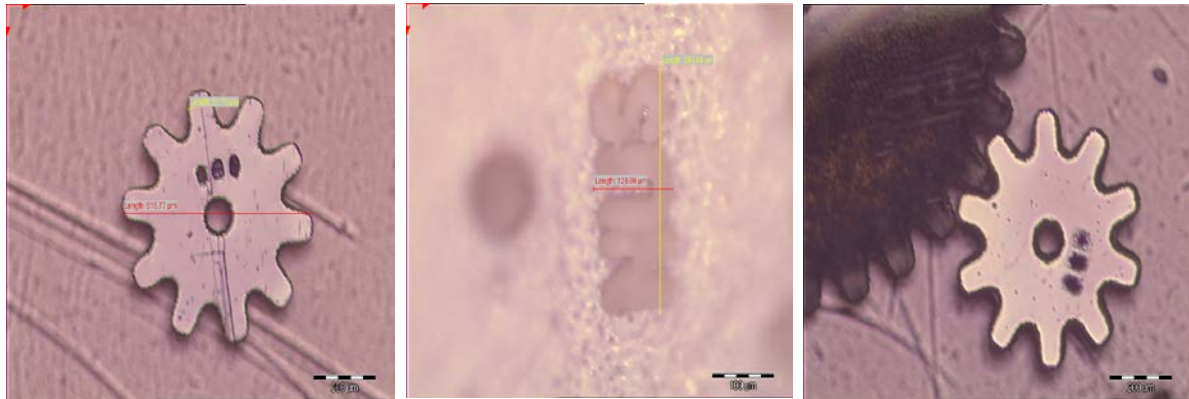


Figure 5.1: Releasing of SU-8 membrane in SU-8 developer and soaking in IPA solution

Special care is required when handling thin SU-8 membrane. If sharp-tip tweezers is used to handle the sample, tearing of the sample may happen. Figure 5.2 shows the optical images of the lifted-off SU-8 micro-gears. Figure 5.3 shows the SEM images of the lifted-off SU-8 micro gears. Figure 5.4 shows the photo-image of the larger size lifted-off SU-8 micro gears.



*Figure 5.2: Optical micrographs of the fabricated micro structure.
The scale represents 100 - 200 μm*

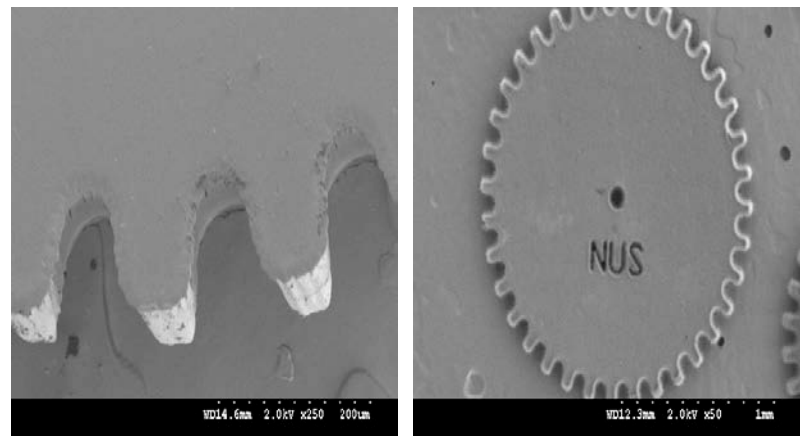


Figure 5.3: Scanning Electron micrographs of the fabricated micro structure



Figure 5.4: Actual [15mm] image of fabricated micro structure

5.1.5 MECHANICAL AND TRIBOLOGICAL TEST RESULTS

Experimental set up was created to study the mechanical properties between the fabricated SU-8 parts with respect to the current method used. Table 5.1 shows the experimental data for mechanical and tribological properties. Figure 5.5 shows the coefficient of friction on the SU-8 release micro structure using ball-on-disk setup with silicon nitride ball as the sliding counterface. The speed of rotation used was 200 RPM with a normal force of 30g. Figure 5.6 (a) shows the wear track optical micrograph obtained from the surface of the SU-8 and Figure 5.6 (b) shows debris collected on the silicon nitride ball because of SU-8 wear. *Singh et al.* [15] reported on the tribological tests carried out on SU-8 thin and thick films. The mechanical and tribological properties of the currently designed process are very identical to those of the existing process. High friction is observed for both processes as SU-8 inherently shows high friction and high adhesion properties. The tribological problems have been largely solved for SU-8 surface [15]. The wear track and the production of wear debris show typical characteristics of SU-8.

<i>Process</i>	Fabricated microstructure	
	<i>Designed process</i>	<i>Existing process</i>
Thickness (μm)	~ 450	~50
Mechanical Properties		
Young's Modulus (GPa)	4.2	4
Hardness (GPa)	0.31	0.35
Tribological Properties		
Water contact angle (degrees)	96	94
Steady state coefficient of friction	0.90	0.64

Table 5.1: Experimental data on the material designed and existing process used and tribological properties between designed and existing process

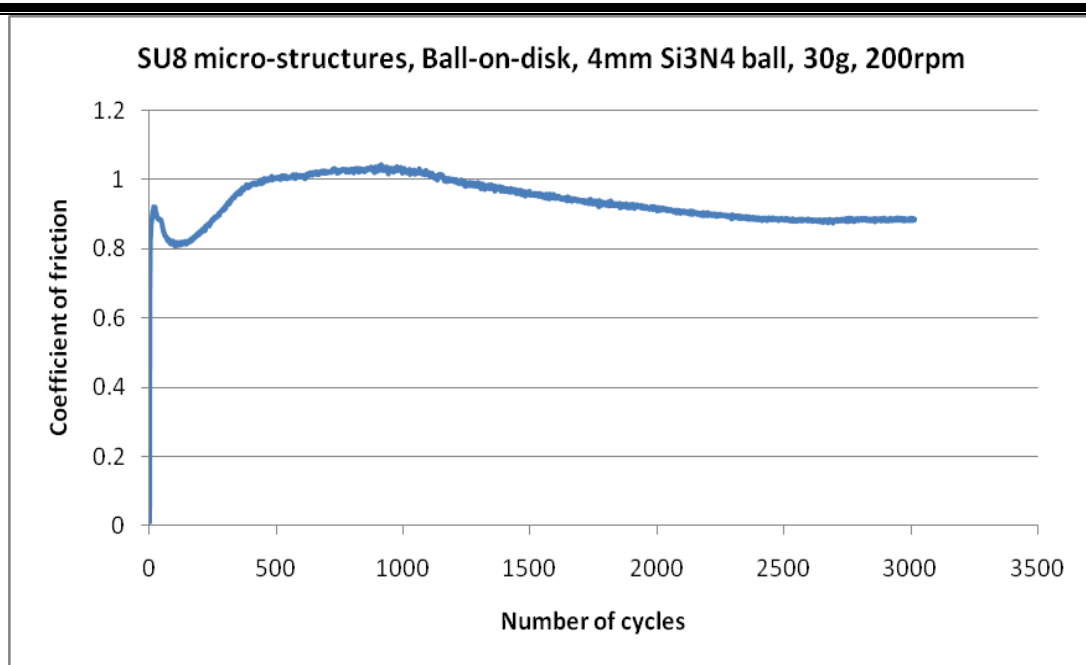


Figure 5.5: Coefficient of friction with respect to the number of cycles on the fabricated structure

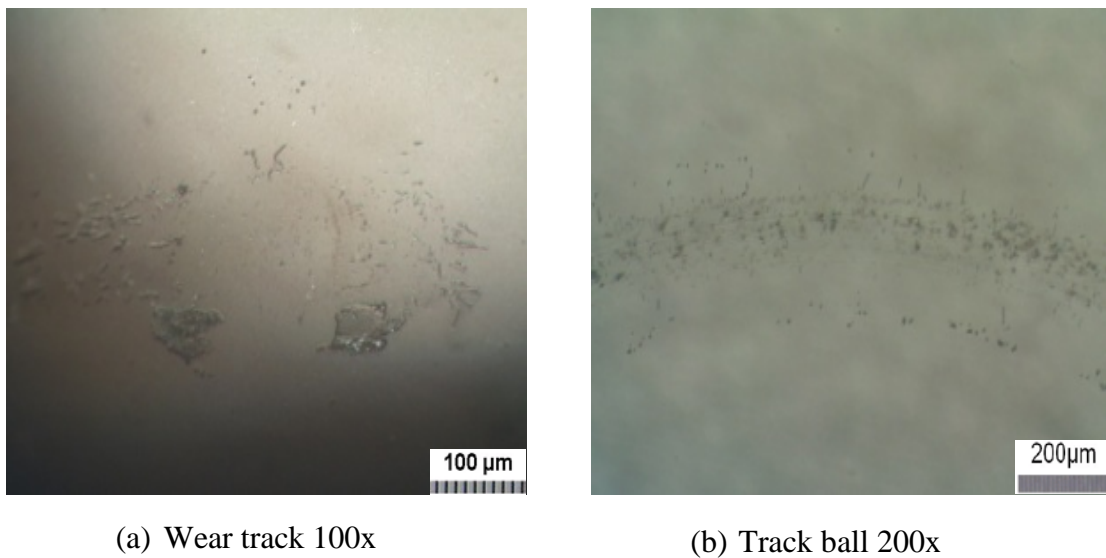


Figure 5.6: Optical micrographs of the wear track on the (a) fabricated structure and (b) Interface ball surface

5.1.7 SUMMARY

This study has proved successfully the initial concept of using positive photo sensitive resist (AZ4620) as the sacrificial layer to lift-off the device layer coated above this layer. There are few key points needed to be taken care of. In order to achieve thicker layer of coat on the device layer, we may require thicker sacrificial layer correspondingly. However, this may result in wasting of material for sacrificial layer. On the other hand, if the thickness is insufficient the lift-off process may not be successful, as the etchant or developer needed to remove away the sacrificial layer can't penetrate through the narrow gap and reach to the center area of the of entire SU-8 layer.

Ultra-violent (UV) dosage is also another factor which needs to be optimized. If the energy dosage is too much, it may cause the surface of SU-8 which is located at the interface between the sacrificial layer to form micro-bubble or cavities. And this may affect it's mechanical performances. Cracks may form resulted from fatigue generated by those micro cavities. In order to resolve this issue, the baking process after SU-8 is coated should to be long enough in order to drive away the solvent embedded within SU-8 resist.

Lastly, in order to reduce the internal stresses of the SU-8 film, sufficient hard-baking of the layer are required after developing is completed. However, over-baking may result in cracking and failure too. Therefore, heat treatment process of SU-8 is also important to be taken note of as this affects the material integrity eventually.

5.2 ENHANCE DEVELOPMENT OF SU-8 STRUCTURES

As reported in Section of 5.1, it clearly shows that use of photoresist as sacrificial layer for lifting off SU-8 can be achieved. However, there are two limitations that need to be highlighted. From the findings, the duration of lifting-off could be long, in the range of 20-30 minutes, if the SU-8 coated area is large. If the length of development exceed the required duration which is considered as safer period, the edge of SU-8 film will tend to warp and degrade the mechanical integrity of the fabricated structure. Therefore, in order to overcome this issue, special improvement process can be used to enhance the initial results obtained. As mentioned, there are basically two types of release technique in polymer MEMS; dry and wet releasing methods. Dry release aims at using low free energy films (SAMS), fluropolymers like Teflon, which reduces the adhesion between the substrate and microstructure can be used. In wet release technique, several sacrificial layers like polystyrene, gold, aluminum, copper are used. The structure release of aluminum is about 160nm/min and would take several hours for release depending on the surface area of the structure. In fabrication of SAMS, toxic silane treatments are required which needs special safety precautions. Plasma deposited fluorocarbon films with low free surface energy which are generally used as anti-stiction layers and hydrophobic coatings on scanning probe microscope to reduce friction and adhesion. But again these depositions require advanced Si etch device which makes the process more expensive and complicated. OmnicoatTM which was used as sacrificial layer (in nanometer range) has a disadvantage of not being used to remove large functional structures by etching. The thickness of OmnicoatTM less than 100nm provides very small gap for the developer and does not allow the dissolution of layer below the

extended structure. Using metal as sacrificial layer such as Cr,Cu,Cr/Cu/Cr,Al is time consuming and expensive and may not be suitable for large structures. Some papers have reported polystyrene used as sacrificial layer. Another dry release technique is reported, in which a fluorocarbon film is used to lift-off SU-8 structures. The fluorocarbon is deposited on the substrate using advanced silicon dry etch device. All these processes involve sophisticated equipments which are not cost effective from commercial point of view.

5.2.1 USING CURRENT LIFT-OFF METHOD FOR SU-8 FILM

In the current process, a positive resist (AZ P4620) is used as a sacrificial layer which acts as a separation between the silicon substrate and SU-8 resist. During developing of SU-8, AZ 4620 resist is also attacked by the developer and dissolved, thus producing a free standing SU-8 structure.

Adhesion force between the silicon and SU-8 will be decreased when thicker sacrificial layer is deployed. However, the stress developed at the Si-SU-8 interface during cross-linking causes the layer to peel off. As a result, the stress is induced because of the volume shrinkage of 7.5% of SU-8 due to different thermal expansion coefficient. This stress increases with increase in lateral dimension and height of the SU-8 structure. The use of photoresist reduces the processing steps and cost. Moreover the sacrificial layer thickness can be controlled depending upon the structural layer thickness which cannot be done when using other processes. The number of masks required is reduced. And once again, this decreases the number of processing steps and ultimately the cost. Table 5.2 shows the experimental results

obtained from the test done to study the durations effect of UV exposure on the different thickness of SU-8 layers coated.

No.	No. of AZ layers	AZ thickness	No. of AZ layers	SU-8 thickness (μm)	Exposure duration(Sec)	Development time (Minutes)
1	7	56	1	50	22	25
2	3	60	2	70	15 (multiple)	20
3	3	50	1	150	120	30
4	1	10	1	165	45	No lift-off
5	1	10	1	165	60	No lift-off
6	2	70	1	165	30	50 – 60

Table 5.2: Experimental results obtained from the test done to study the duration's effect of UV exposure on the different thickness of SU-8 layers coated

It could be observed that as the SU-8 structure thickness increases, the sacrificial layer thickness should also increase for it to get fully lifted off. The SU-8 structures were able to lift off only when nearly equal thickness of AZ was coated before SU-8. This is a slight drawback as the process time gets increased and thus the quality of structure gets affected. For a thinner AZ layer, the developer could not penetrate through the sides of SU-8 and dissolve the sacrificial layer hence the structure strongly adheres to the silicon wafer and a large mechanical force has to be applied on the SU-8 in order to peel-off and this eventually leads to damage to the structure. It was also observed that during Post Exposure baking, air voids were formed on the surface of the SU-8 structure which was due to the insufficient soft baking. Also, the solvent from the underneath AZ layer contributes to the bubble formation as the SU-8 is directly coated on top of it. Over exposure of SU-8 results in cracks in SU-8

layer. Under exposure has a tendency to undercut the SU-8 structure. When fabricating very thick SU-8 structures, the main problem is the development of internal stress. This stress leads to cracking and shrinkage of the patterned structure. The following picture depicts the crack formation, adhesion and bubble formation due to disturbances in the process. Figure 5.7 shows the photographs of (a) Bubble formation on the UV exposure region after post exposure baking (PEB) process and (b) Shrinkage effect due to overexposure with stress formation within the SU-8 film

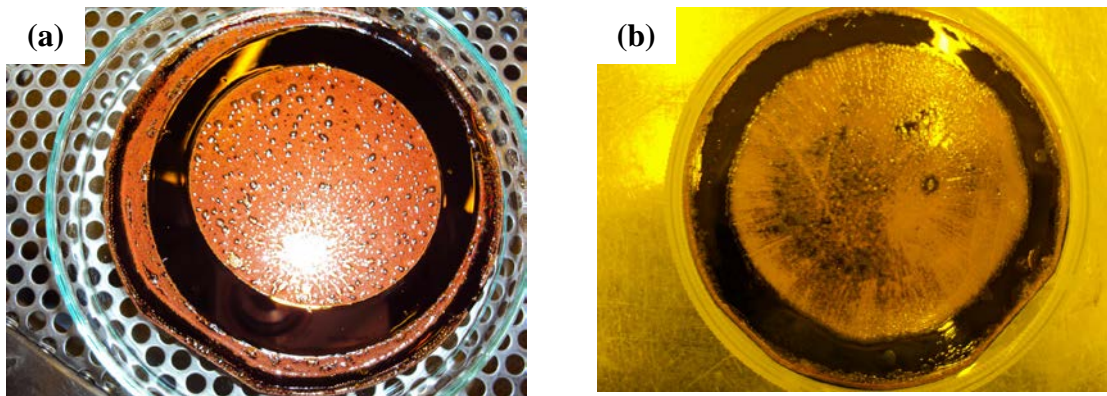


Figure 5.7: Photographs of (a) Bubble formation on the UV exposure region after post exposure baking (PEB) process and (b) Shrinkage effect due to overexposure with stress formation in within the SU-8 film

Figure 5.7 shows the lifted-off sample of SU-8 film after developing process. Micrographs of lifted SU-8 film surface were also examined. Figure 5.8 shows (a) & (b) photographs of 160 μm thick SU-8 film after lift-off, (c) photographs of 160 μm thick SU-8 film adhesive onto 10 μm AZ resist thickness and silicon substrate, (d) micrographs of air void trapped in between AZ resist and SU-8 layer, (e) micrographs of SU-8 layer after UV exposure which is over-exposed and (f) micrographs of surface of SU-8 microstructure after development process.

In order to further solve the lift-off problem and reduce the lift-off duration, we propose to use a surface modified silicon substrate before a deposition of the AZ

resist sacrificial layer. This type of surface modification should aim at reducing the adhesion and hence the surface energy between the silicon substrate and the coated layers.

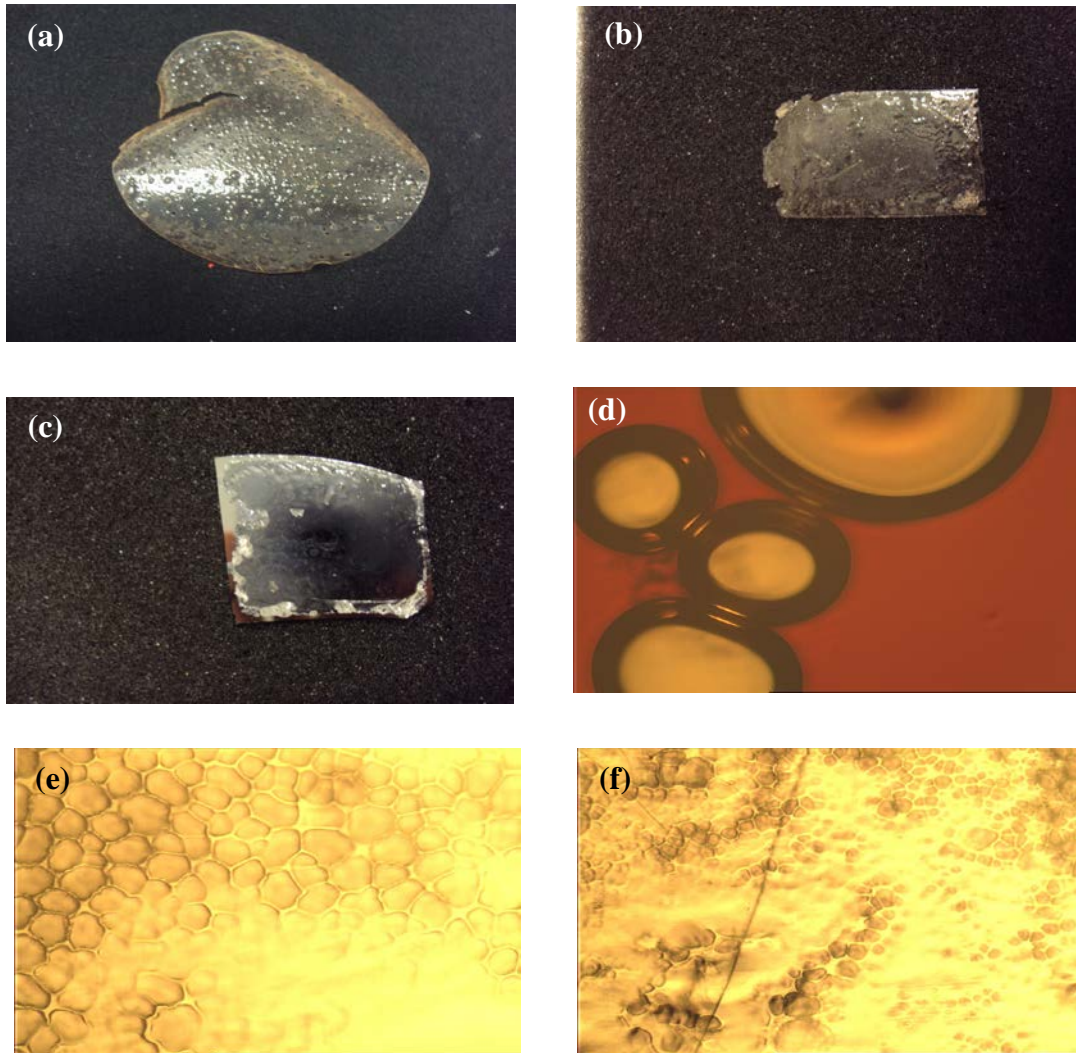


Figure 5.8: Photographs and micrographs of lift-off SU-8 film and surface examination of SU-8 film (a) Larger area of lifted SU-8 film with warping edge (b) Smaller area of lifted SU-8 film without warpage (c) SU-8 film adhesive onto silicon substrate with AZ layer (d) Bubble formation on the surface of SU-8 film adhesive onto silicon substrate (e) Backside surface of lift-off SU-8 film (f) Front side surface of lift-off SU-8 film

5.2.2 USING METALLIC ENHANCEMENT LAYER FOR LIFT-OFF PROCESS

As mentioned in the previous section (5.2.1), problems encountered made the current lift-off process slightly more difficult in realizing the lift-off of larger SU-8 structures. The reason for this was investigated by finding out the surface energy of various combinations of substrate samples. The surface energy was measured by acid-base method where in 2 polar and 2 non polar liquids are used and their contact angles with coated samples are observed. Table 5.3 provides surface energy data for the samples. The surface energies presented are for silicon, silicon with oxygen plasma, silicon with gold coating only, silicon with aluminium, silicon with copper and silicon with chromium and gold coated. Contact angles and surface free energies of different specimens were determined by VCA Optima Contact angle System (AST product, Inc., USA).

Lift-off duration for the whole SU-8 sample was observed and recorded. From observation, the whole SU-8 film with approximately 100 micron thickness will be able to lift off successfully at the shortest timing of 30 minutes which is located at the centre of the substrate. However, those samples located at the edge of the wafer will tend to lift-off at a much longer process duration. These tests were carried out without the assistance of ultrasonic agitation. In order to shorten the lift-off duration, special coating needed to be deposited on the surface of the silicon substrate before over-coating with the sacrificial layer of AZ 4620 positive photoresist. The selection of the coating must be aimed at reducing the surface free energy of the substrate silicon. With the extra coating of this particular layer, the sacrificial

RESULTS AND DISCUSSION

layer thickness could also be reduced. And this can provide advantages in term of speeding up the whole fabrication process and reducing cost of material. Figure 5.9 shows the image of water contact angle of different specimens.

Sample Solvent	No. of tests	Silicon	Silicon + O ₂ plasma treatment	Silicon + gold	Silicon + Aluminum	Silicon + Copper	Silicon + Chromium+gold
DI water	1	33.6	9.1	83	90.3	80.3	107.3
	2	29.4	5.7	81.9	96.2	87.3	104.9
	3	31	12.8	81.4	100.8	91.6	90
Ethanol	1	12.3	10.6	6	11.2	11.8	75.3
	2	11.1	9	7	14.1	11.6	81.2
	3	10.9	5.6	8.8	13.8	8.2	80.3
Xylene	1	56	36.1	6.8	85.4	17.9	66.2
	2	48.8	51.6	11.3	87	12.5	67.2
	3	50.3	43.1	8.2	85	10.8	69.5
Hexadecane	1	9.1	7.7	92.6	5.1	100	82.2
	2	11.9	11.2	92.5	3.5	100.8	81.2
	3	9.7	8.3	104.2	4.5	98.8	74.9
Surface Free Energy, dyne/cm		67.18	56.2	33.76	21.31	23.7	13.58

Table 5.3: Surface free energy measurement of different specimens

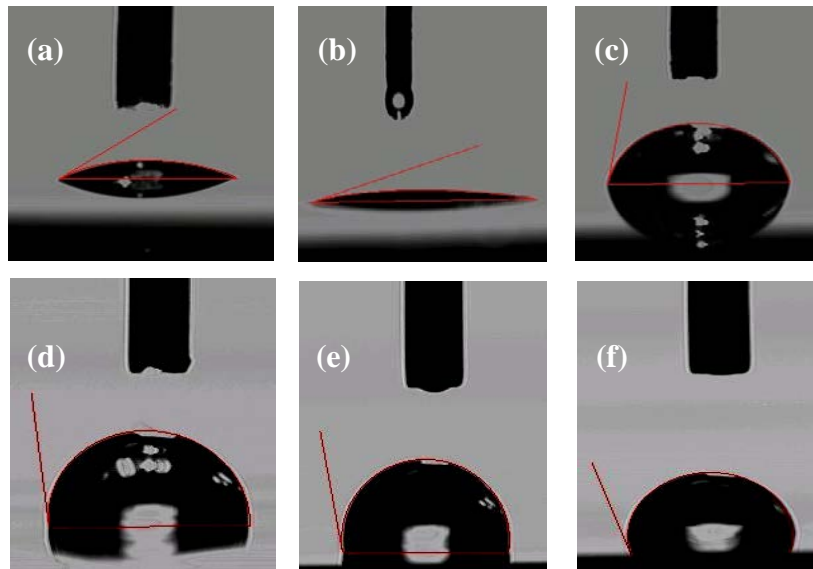


Figure 5.9: Water contact angle image of (a) Bare Si, (b) Si + O₂ plasma, (c) Si + Au (Sputtered), (d) Si + Al (Sputtered), (e) Si + Cu (Sputtered) and (f) Si + Cr + Au (Evaporation)

Surface free energy tests were also carried out to find out the surface free energy of AZ 4620 positive photo-resist and SU-8 coated layers where both materials were coated onto the silicon substrate. Table 5.4 shows the results of surface energy obtained for AZ 4620 without UV exposure, AZ 4620 with UV exposure, SU-8 without UV exposure and SU-8 with UV exposure. Figure 5.10 shows the water droplet images of the resist and SU-8.

Sample Solvent	No. of tests	AZ 4620 without UV exposure	AZ 4620 with UV exposure	SU-8 without UV exposure	SU-8 with UV exposure
DI water	1	119.4	80.3	66	69.7
	2	109.6	84.7	68.1	73.6
	3	119.8	90.3	69.3	76.1
Ethanol	1	21.9	14	4.3	8.1
	2	22.4	17.3	10.9	7.5
	3	22.2	11	10.9	12.6
Xylene	1	16.3	13.8	15.7	9.9
	2	22.5	18.3	14.5	9.1
	3	18.7	10.5	11.1	10.6
Hexadecane	1	20.3	39	34.2	42.2
	2	24.7	33.1	31.7	34.8
	3	23.7	37.5	31.8	37.2
Surface Free Energy, dyne/cm		26.06	33.47	38.14	33.58

Table 5.4: Surface energy obtained for AZ 4620 without UV exposure, AZ 4620 with UV exposure, SU-8 without UV exposure and SU-8 with UV exposure

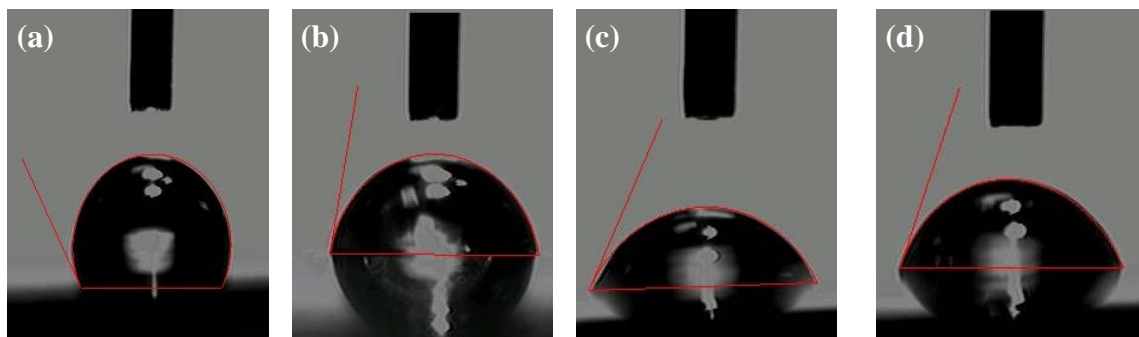


Figure 5.10: Water contact angle image of (a) AZ 4620 without UV exposure, (b) AZ 4620 with UV exposure, (c) SU-8 without UV exposure and (d) SU-8 with UV exposure

From the data shown it can be seen that the surface energy of bare silicon is very high (67.18 dyne/cm) as compared to that of other samples. This proves that the surface of silicon is hydrophilic. However, this energy is very high in comparison to that of AZ 4620 photoresist which is 33.47 dyne/cm and SU-8 which is 33.58 dyne/cm. The differences in surface energy between the substrate and the coated layers are very large thus making the adhesion very significant. In this case, the AZ layer which has low surface energy forms a film on the silicon substrate by consuming energy from the substrate. The greater the energy of the source (substrate), the greater will be the bonding force between the film and the substrate. This bonding effect will cause difficulty in the lifting off process as a force/ agitation greater than this bonding force is required to separate the SU-8 from silicon surface. The traditional way to solve this problem is to have thicker coatings of the sacrificial layers. Below shows Young's equation to calculate the energy balance of a water droplet on a solid surface which is expressed as

$$\gamma_{LG} \cos \theta_c = \gamma_{SG} - \gamma_{SL}$$

Where

- γ_{SL} is interfacial tension between solid and liquid
- θ_c is the equilibrium contact angle of a drop of water
- γ_{LG} is surface energy of liquid with the units of (mJ/m²)
- γ_{SG} is interfacial tension between solid and vapor

This interfacial energy difference between the liquid and solid should be maintained low so that the adhesion is minimized.

5.2.3 SOLUTION AND NEW METHODOLOGY

From the above experimental investigation conducted, it is clearly shown that we can modify the surface property of the silicon surface by various surface modifications. Low surface free energy would be beneficial for low adhesion. The sacrificial layer or the substrate should have low surface energy so as to decrease the adhesive force. At the same time, the surface should have sufficient surface energy for spin coating of resist to be carried out without the problem of de-wetting. Obviously, this would require an optimization of the surface free energy of the substrate.

In order to avoid the stiction issue due to wet release during development, the surface tension forces are reduced by employing metal base layer and sacrificial layer which acts as a double protection to the SU-8 layer which give rise to less interactive forces between the substrate and the resist. Coatings of metals such as Gold, Aluminum, Copper, and Chromium are used in our experiment in order to determine their efficiency in the lift-off process. A sample of piece of bare Si wafer was Au sputtered using sputtering system for 10 minutes at 30mA, 10 mbar pressures. The contact angle measurement reveals that the surface energy of the sample is about 33.76dyne/cm which is very close to the AZ film value. Thus the difference in surface energy is reduced drastically from 33.71dyne/cm to 0.28dyne/cm. This is a good sign which can ease the lift-off process.

There are several advantages of our method. One of them is that, here the metal base coating is not disturbed during the process since AZ layer is coated above

it and this acts as sacrificial layer and not the metal coating. The metal coated base can be re-used again which saves the cost of production when considered in bulk.

When the separation between Si and SU-8 is increased by thicker sacrificial layer, SU-8 can be easily peeled off with the stress developed in within SU-8 layer during the cross-linking process which creates stress at the Si-SU-8 interface. This stress increases with lateral dimension and height of the structure. The use of metal base layer reduces the requirement of coating thick sacrificial layer and hence reduces the processing time to a greater extent. Moreover, thin sacrificial layer can be used for thick SU-8 structures which are very advantageous. Similarly, the extended pre-baking increases the mobility of the polymer molecules. Ramping of temperature under the glass transition temperature allows the polymer molecule to recrystallize in a stress free way. During the fabrication, the following parameters are maintained so as to obtain a uniform result.

- After every spin coating, a rest period of 10 minutes was kept for the resist to distribute evenly on the substrate.
- The substrate is rotated at 20 rpm while dispensing SU-8 to give an even distribution of resist on silicon wafer.
- The spin speed was ramped up slowly from 500 rpm to the desired value
- Similarly the soft baking is done by ramping up the temperature slowly so that the polymer molecules re-crystallize in a stress free way.
- Sample is cooled after every baking cycle for minimum 10 min to relieve the thermal stress developed during baking.
- The exposure dosage of 40sec is split thrice with a dwell period of 10 sec and exposure period of 13 sec.

- Ultra sonic bath is used to provide agitation during development of the sample.

Multiple exposures were beneficial since the dwell period allowed the resist to absorb the energy completely which greatly influenced the quality of the structure.

Figure 5.11 shows the photographs of (a) SU-8 pattern on bare silicon wafer over-coated with thin layer of AZ resist and (b) SU-8 film during development.

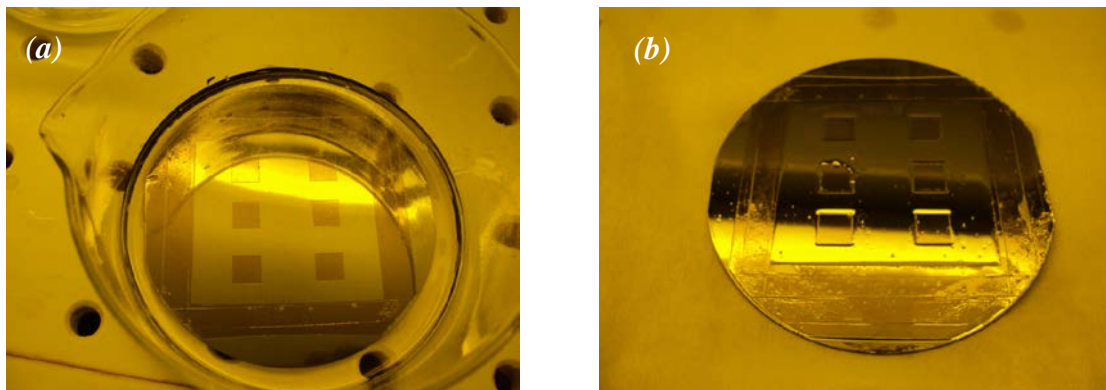


Figure 5.11 shows the photographs of (a) SU-8 pattern on bare silicon wafer over-coated with thin layer of AZ resist and (b) SU-8 film during development

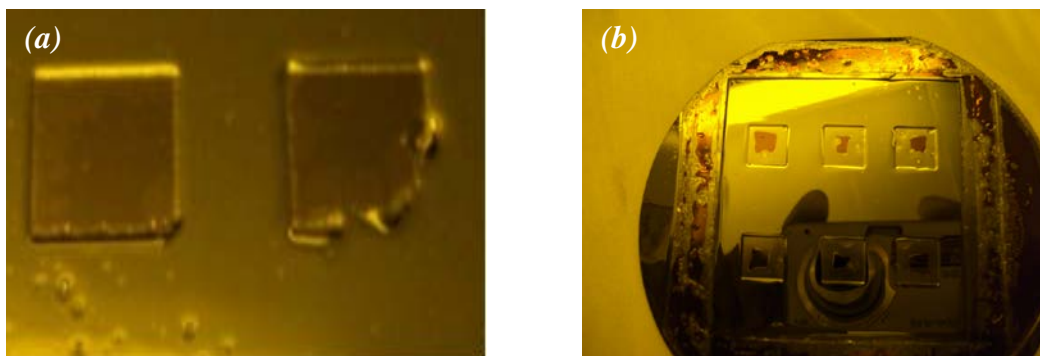


Figure 5.12 shows the photographs of (a) Distorted SU-8 structure on bare silicon wafer and (b) SU-8 film during development using thick film AZ on bare silicon wafer

It was observed (shown in Figure 5.12 a) that the thin film AZ coated sample was not developed even after 40 minutes in the ultrasonic bath. The AZ layer beneath the SU-8 was not attacked by the developer. Even though SU-8 is separated from the substrate by AZ photoresist layer, the adhesion was still observed to be strong

enough. The reason could be that the bottom surface of the AZ interacts strongly with the silicon substrate and the top surface is attached to SU-8 which prevents the structure from peeling off easily. When the thickness of AZ was increased to 30 μm , the structures were released within 15 minutes which can be seen from the above image (Figure 5.13a and 5.13b)

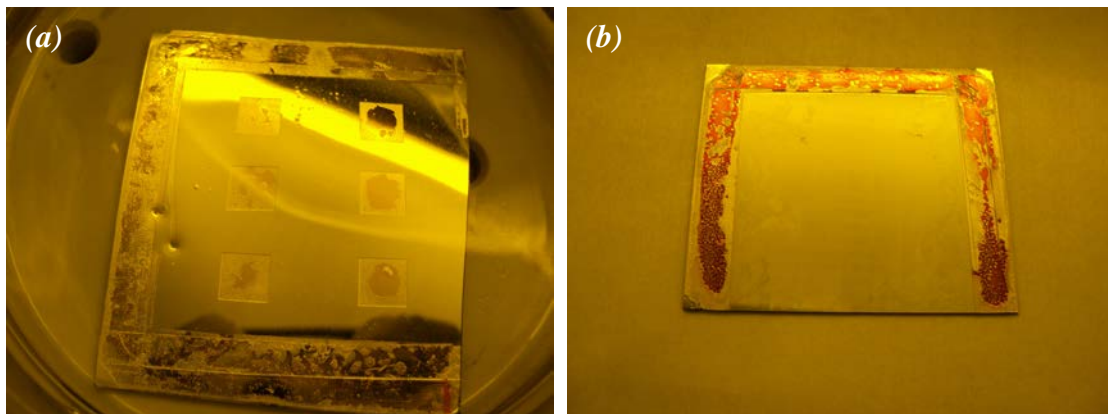


Figure 5.13: Photoimage taken (a) during development and lift-off process with SU-8 developer with SU-8 structure coated on aluminum surface and (b) after completion of lift-off process after 2 minutes.

On the other hand, when an aluminium coated Si substrate was used, the structures developed at a very fast rate of about within 8 minutes (for thin film AZ) and 2.5 minutes (for thick film AZ). This rate of lift-off is much shorter period so far reported in the literature. Even a thinner AZ layer would suffice the lift-off requirement when used with aluminum metallized silicon substrate. This is a major advantage in terms of time and cost.

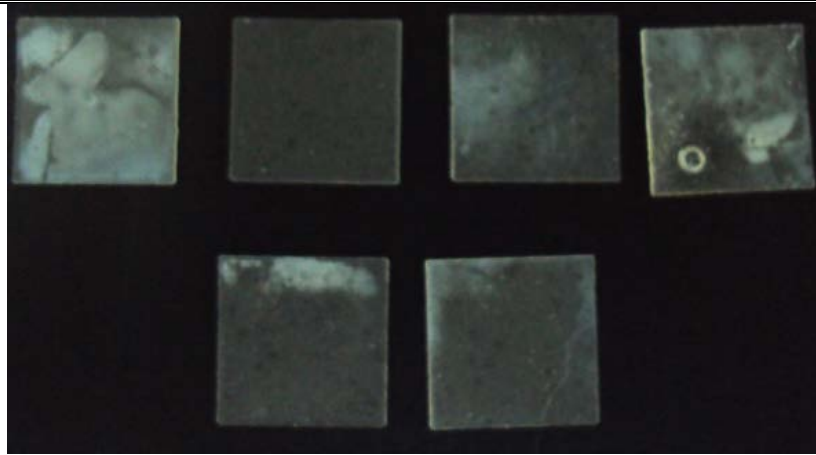


Figure 5.14: Photoimage taken for SU-8 lifted off film using the process of aluminum coated surface together with AZ photoresist as sacrificial layer.

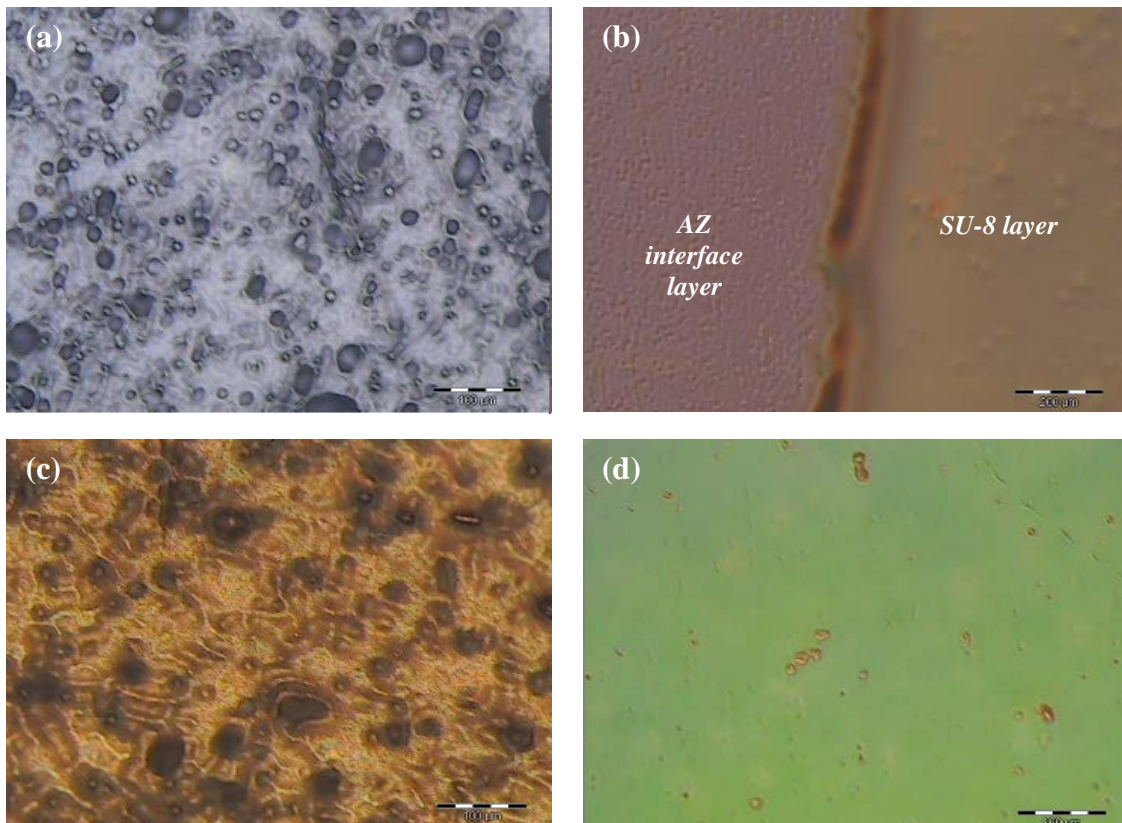
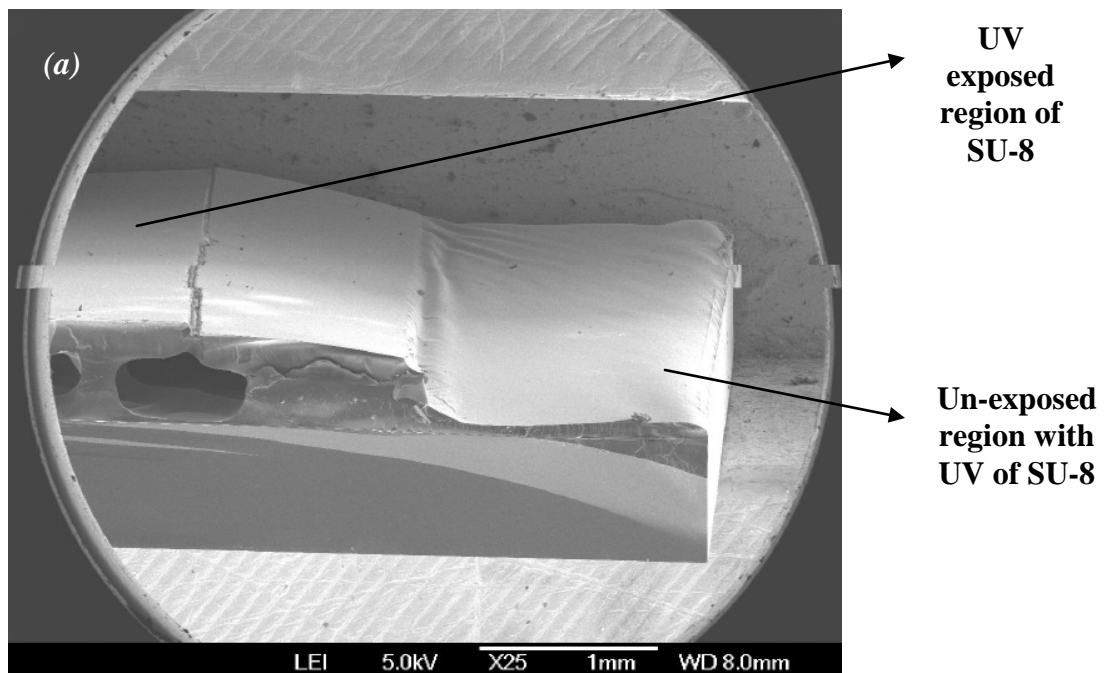


Figure 5.15: Micrographs taken for lifted-off SU-8 film using (a) Top surface of SU-8 with UV exposed using normal lift-off method with AZ positive photoresist as sacrificial layer, (b) SU-8 layer with AZ positive photoresist interface layer, (c) Bottom surface of SU-8 with UV exposed using normal lift-off method with metallic base material for enhance lift-off process and (d) Top surface of SU-8 UV exposed surface with metal base sample

Figure 5.15 shows micrographs taken for each methods used for lift-off process on SU-8 surfaces. From these micrographs obtained, we could observe a decrease in granular concentration between the surfaces. It is evident that surface quality of SU-8 structures with metallized sample is enhanced compared to the only AZ resist method. One of the reasons attributed for this would be that the dual coating of metal and resist provides smoother and even surfaces compared to unmodified silicon samples. By this means we are able to fabricate SU-8 based MEMS structures with much better quality. Figure 5.16a shows the cross-sectional scanning electron microscopy image of UV exposed and non-exposed region for SU-8 film. Figure 5.16b shows the cross-sectional scanning electron microscopy image of the details of each individual layer coated.



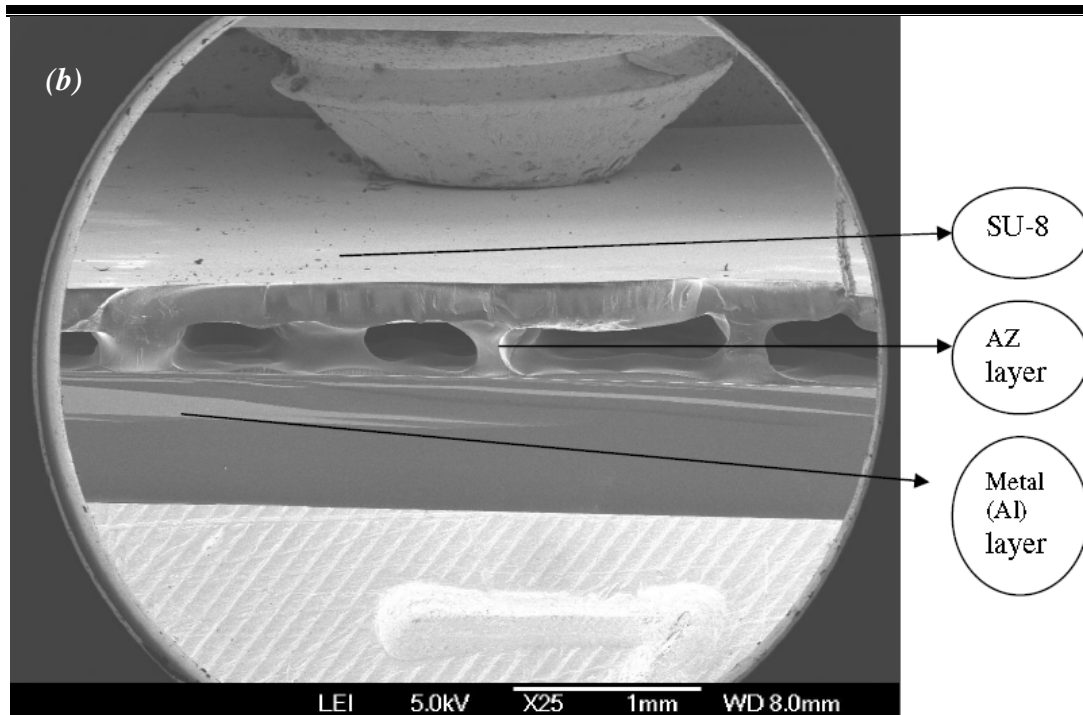


Figure 5.16 (a) show the cross-sectional scanning electron microscopy image of UV expose and non expose region for SU-8 film and (b) show the cross-sectional scanning electron microscopy image of the detail of each individual layer coated.

From the cross-sectional SEM image obtained, we could observe the voids in the AZ layer which is sandwiched between aluminium coating and SU-8 layer. This void in a way helps in providing passage to the developer to seep through the bottom of SU-8 and dissolve the sacrificial layer. This in turn reduces the development time to a greater extent.

5.2.4 FABRICATION OF MICRO TIPS STRUCTURE USING THE CURRENT LIFT-OFF METHOD

After developing the enhance technique on SU-8 lift-off, another idea of fabricating micro tips structures was formed. As reported by *Taff et al.* [39], colour mask can be used instead of gray scale mask to produce three dimensional micro structures. Previously, gray scale mask has been used to fabricate 3D structure. However, the cost of producing gray scale mask is too expensive. Therefore, *Taff et al.* came up with a method of using colour mask produced by laser colour printout on a transparency instead of on a glass mask. Figure 5.17 shows the colour masks produced using laser colour printer on transparency

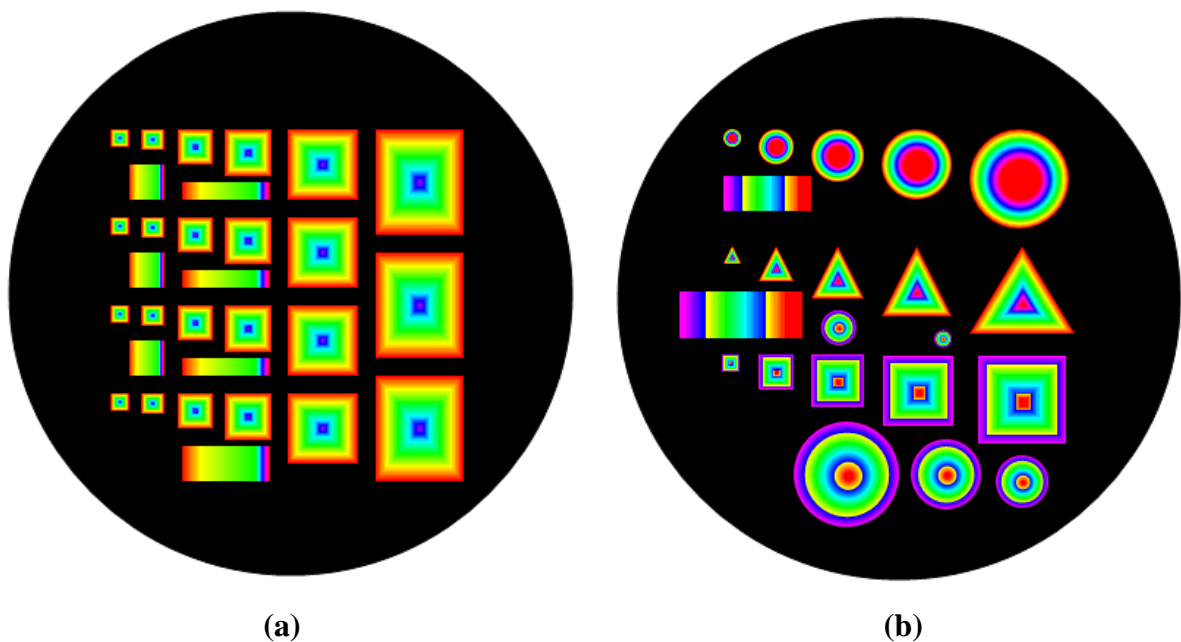


Figure 5.17 Colour masks produced using laser colour printer on transparency with different range of colour

The image in Figure 5.18 shows the exposed SU-8 film developed using SU-8 developer. From the image, protruding layer could be seen. It is to be noted that as the development of the structure will be in steps formation, the cross-linking effect will not be the same for each layers. Therefore, after the development was completed, the sample needs to go through short duration of UV exposure again in order to achieve a full cross-linked result on SU-8 film.

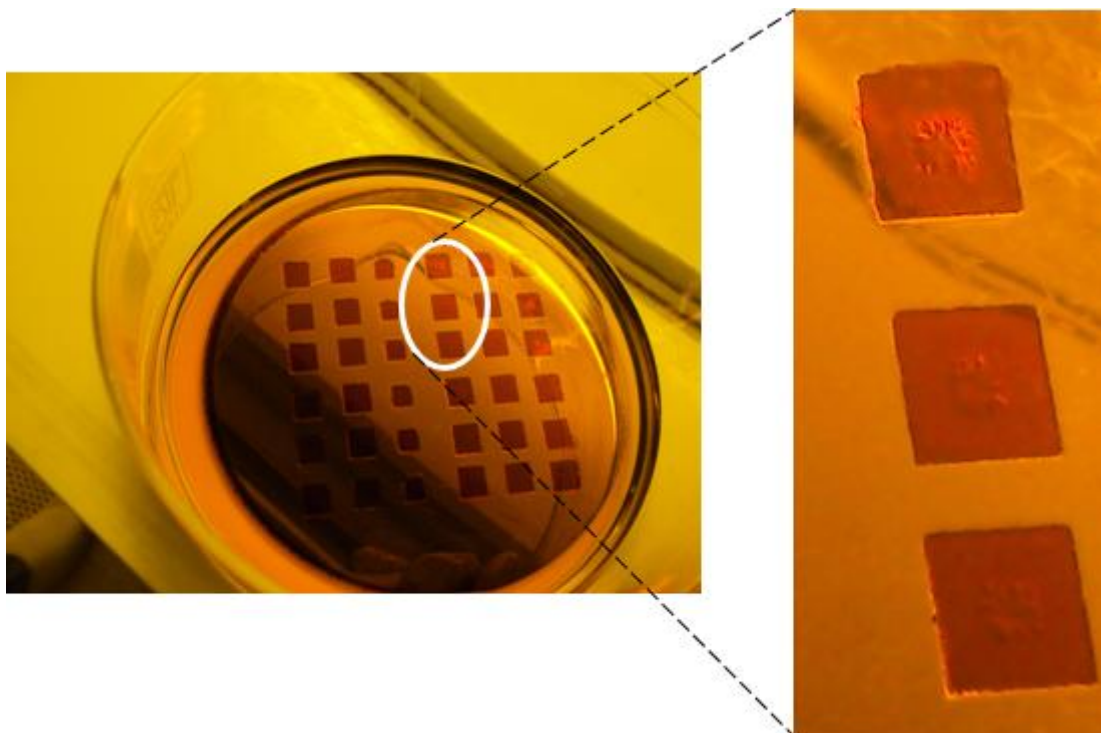


Figure 5.18: Photographs taken during development of 3D SU-8 micro tip structure in developer

SEM imaging was also conducted to see the protruding area of the SU-8 film. From the image (Figure 5.19), slight protruding region can be seen and that show that the effect of colour is workable in combination with the current method of lift-off. Figure 5.20 shows the surface profiling result obtained using a stylus profiler system. From the surface profiling measurement obtained, it is shown that different colour tone will affect the height of the SU-8 fabricated. Different colour tone will block the

amount of UV passing through the mask and affect the height of the SU-8 film developed. It is obvious that with some optimization of the mask layout, much sharper and/or complex shaped features can be made with SU-8 when the AZ resist is used as the sacrificial layer.

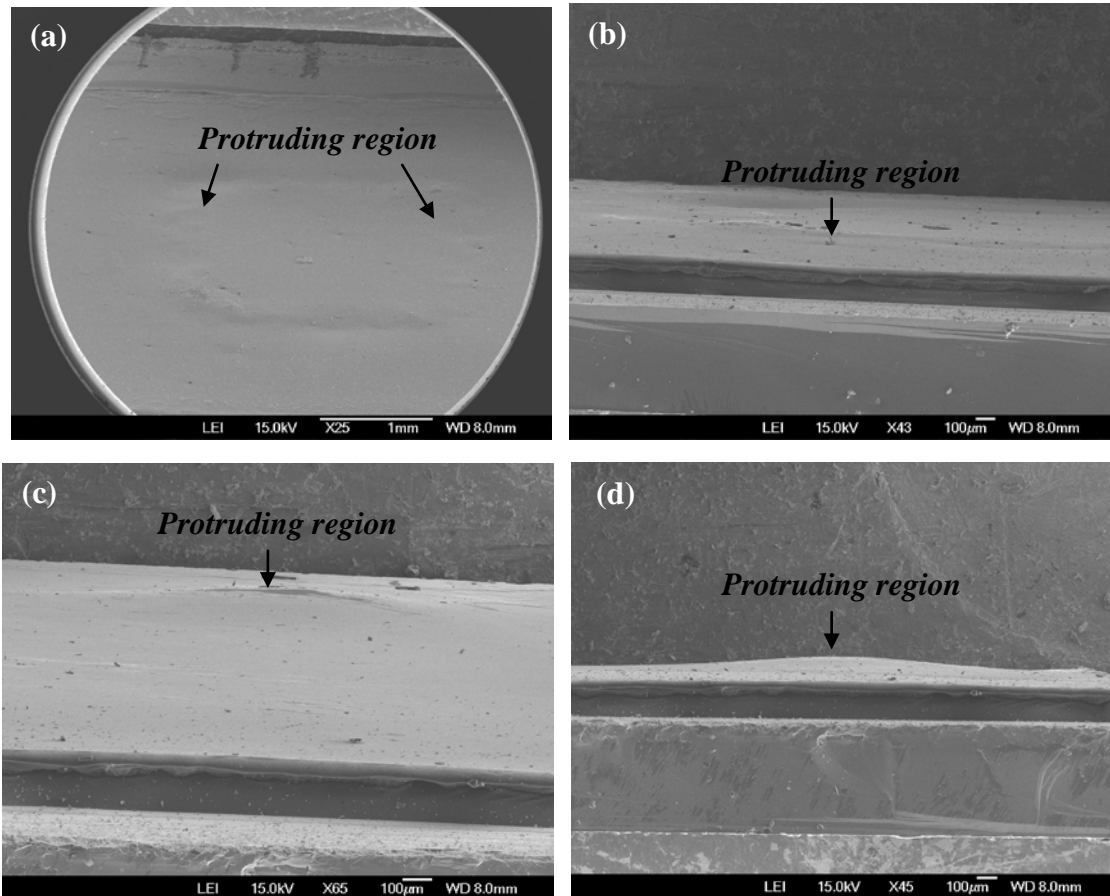


Figure 5.19: Cross-section SEM micrographs for (a) Wide viewing magnification, (b) Tilted at 10° (c) Tilted at 20° and (d) Tilted at 90°

This method is a very cost-effective and time-effective means of producing SU-8 MEMS.

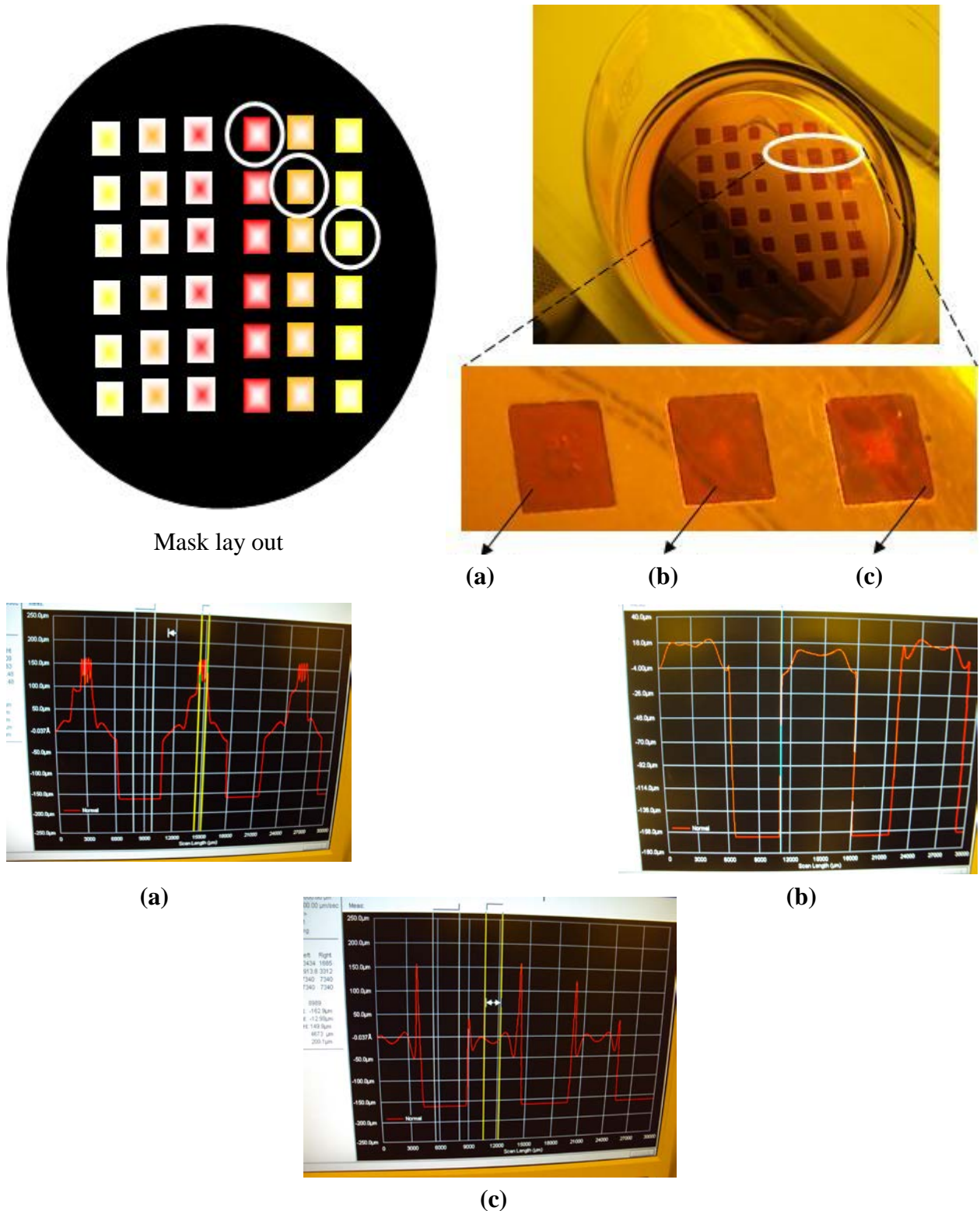


Figure 5.20: Surface profiling result obtained using a stylus profiler system on three different colour tones

CHAPTER 6 – CONCLUSIONS

The project has successfully created SU-8 micro-structure by using AZ 4620 positive photo-resist as the sacrificial layer with the aid of lift-off technique. SU-8 film with thickness ranging from 10 to 500 micrometer can be achieved. Mechanical property characterization and tribological analysis are carried out in order to make comparison with the SU-8 sample prepared by normal LIGA fabrication and the method developed in the current study. From the results obtained, it can be concluded that the present method gives similar results in terms of the mechanical properties of the fabricated micro components.

The AZ photoresist can be applied by spin-coating in several layers on silicon substrate to be used as a sacrificial layer for SU-8 lift-off. The thickness of AZ layer will depend upon the thickness of SU-8 layer. The AZ layer thickness will also decide the time duration of lift-off which may vary from 20-30 minutes. A longer lift-off process may cause edge of the SU-8 structure to warp as stress will start to form near the edges.

In order to further reduce the lift-off time and improve the quality of the SU-8 structures, the surface energy of the silicon substrate was optimized by metallization. It has been shown in this study that modifying silicon surface with a layer of aluminium can help reduce the lift-off time to only 8 minutes for thick AZ layer and 2.5 minutes for thin AZ layer as compared to 20-30 minutes for only AZ layer. This presents great savings in time to fabricate SU-8 micro-structures.

Finally, the current novel SU-8 lift-off technology was applied to the fabrication of a tip made of SU-8. The study proves that the current lift-off method is capable of fabricating 3D structures such as tips and gears.

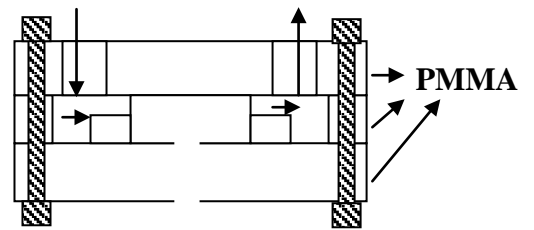
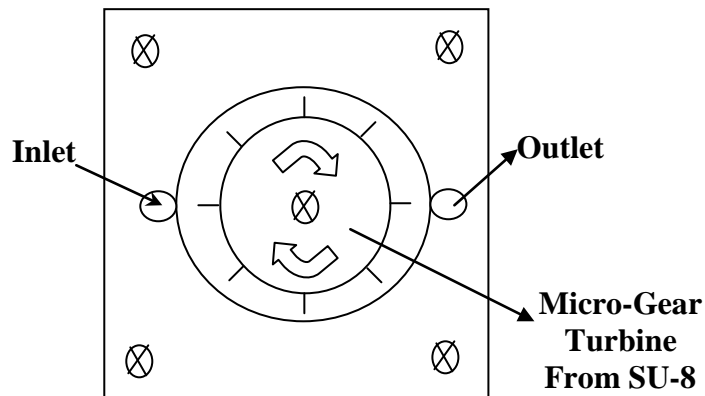
CHAPTER 7 – FUTURE WORK

7.1 ADDITION OF NANO-PARTICLES INTO SU-8 FILM

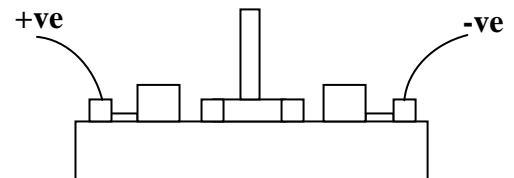
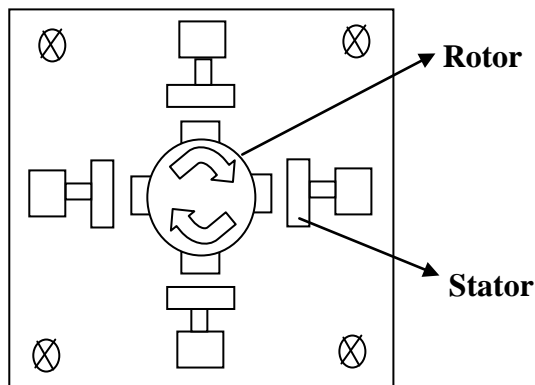
In order to produce SU-8 film with better functionality in terms of mechanical and electrical properties, specific nano-particles could be added into the SU-8 as a mixture. Electrically enabled SU-8 micro structure can be fabricated. Currently comb-drive is fabricated with silicon as the material; it is not common in using polymer as material as it is insulator in nature. However, if conducting nano-particles are mixed with SU-8, this idea could be experimented. And further development of the lift-off technique could be carried out as SU-8 mixed with nano-particles will be different from those with just SU-8 only.

7.2 DEVICE LEVEL FABRICATION WITH FULL INTEGRATION OF LIFT-OFF PROCESS

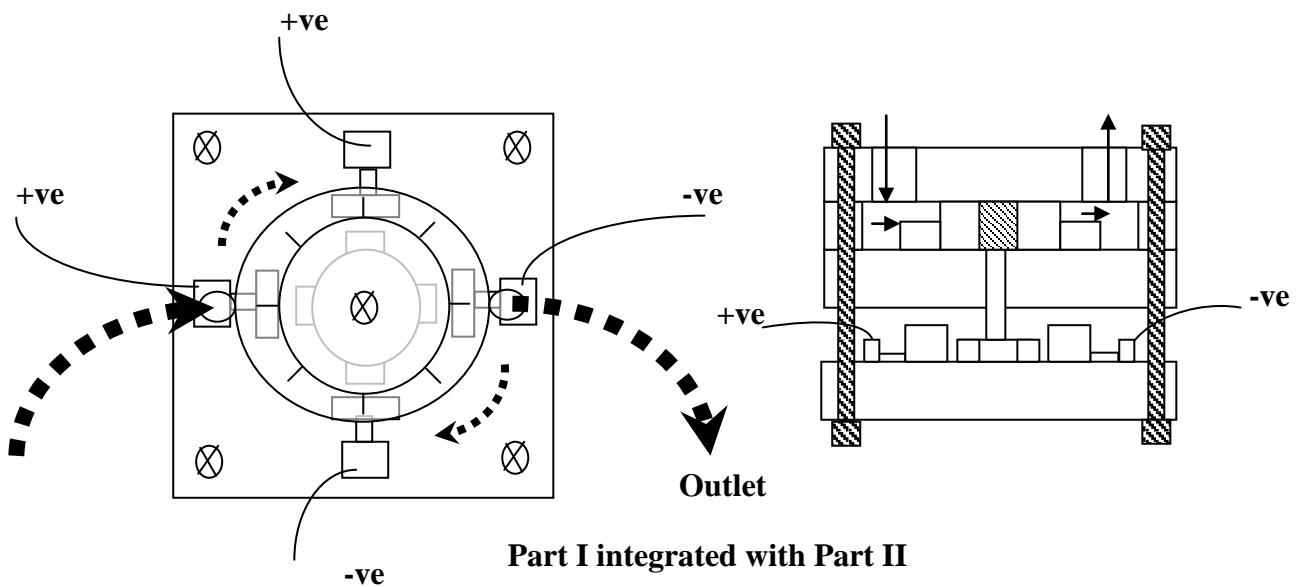
Using the current lift-off method, we can fabricate SU-8 moveable parts and integrate into a MEMS or BioMEMS system in the future. Devices such as micro-pump system, micro actuating system etc can be designed and fabricated using SU-8 material. Figure 8.1 shows the idea of fabrication and integration of micro parts together and form into micro-pump system.



Part I



Part II



Part I integrated with Part II

Figure 7.1 Idea on full integrated micro pump system using SU-8 micro gear turbine

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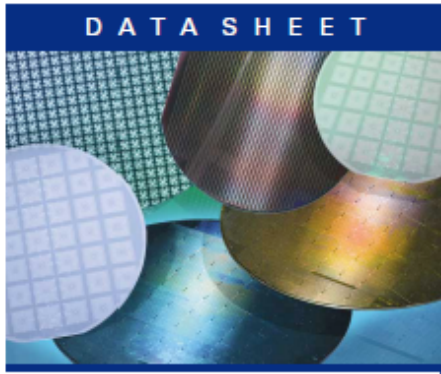
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***APPENDIX A – AZ 4620 POSITIVE PHOTORESIST DATA
SHEET***





AZ[®] P4000 Thick Film Photoresist

Description

AZ[®] P4000 series photoresists provide unmatched capabilities in demanding applications requiring film thicknesses ranging from 3 to over 60 μm . These production proven photoresists set the standard in MR and inductive thin film coil plating, wafer bumping processes, ceramic packaging, air bearing/slider applications and permanent insulation layers. The photoresists can be fully cross-linked to act as a dielectric and remain part of a permanent device structure.

The rapid evolution in the packaging market along with higher resist performance requirements have led to the development of a version of this resist that meets demanding ultra-thick film needs of 60 μm with single coat processes.

Spin, spray, and roller-coat versions of the AZ P4000 series photoresists are available.

Features	Benefits
Steep wall profiles and excellent adhesion on a wide variety of substrates	<ul style="list-style-type: none"> • Ideal for up-plating • No underplating even in thick films
Sensitive to g-, h-, and i-line wavelengths	<ul style="list-style-type: none"> • Sensitive to all popular exposure tools
Available in viscosities that allow coating thicknesses greater than 60 μm	<ul style="list-style-type: none"> • Single resist series that can be used in a wide range of applications
Excellent ion-milling properties	<ul style="list-style-type: none"> • High yields • No cracking, peeling, or bubbling
Exceptionally stable cured films	<ul style="list-style-type: none"> • Provides an excellent, easy to use permanent insulator layer for critical high reliability applications in thin film recording heads
Cast in PGMEA safer solvent with no co-solvent	<ul style="list-style-type: none"> • Toxicity hazard is extremely low • Provides excellent coating properties

Recommended Process

20+ μm Process for AZ[®] P4620 Photoresist: Single coat for track and hotplate
Single coating using either SVG track or Flexifab

Step	Event	Time (sec)	Speed (rpm)	Accel (krpm/sec)
1		4	500	20
2	Dispense resist	5	0	0
3	Spread	3 - 5	300	20
4	"Spike"	0.2	2000	50
5	EBR	20	400	20
6	EBR dry	10	400	20

Bake: Hotplate

Step	Time (sec)	Temp. (°C)	Gap Height (μm)
1	60	120	0.050 (approx. 70°C)
2	60	120	0.025 (approx. 100°C)
3	120 - 180	120	Full contact



AZ® P4000 Thick Film Photoresist

Recommended Process

24 μm Process for AZ® P4620 Photoresist: Double coat for track and hotplate

First Coat: Target 10 μm Film Thickness

Step	Event	Time (sec)	Speed (rpm)	Accel (krpm/sec)
1	SpinLS	2	300	50
2	Dispense resist	10	0	
3	SpinLS	3	300	50
4	SpinHS	60	2500*	50
5	EBR	10	500	50
6	SpinHS	10	1000	50

*Estimated rpm: change for thickness requirements

First Softbake

Step	Event	Time (sec)	Temp. (°C)	Gap Height (μm)
1	Gap*	10	110	0.001
2	Bake	80	110	Full contact

*Gap used to imitate slow heating of substrate.
Use 85 sec bake if gap function not available.

Second Coat: Target 24.0 μm Total Film Thickness

Step	Event	Time (sec)	Speed (rpm)	Accel (krpm/sec)
1	SpinLS	2	300	50
2	Dispense resist	10	0	
3	SpinLS	3	300	50
4	SpinHS	60	1600*	50
5	EBR	10	500	50
6	SpinHS	10	1000	50

*Estimated rpm: change for thickness requirements

Second Softbake: 110°C

Step	Event	Time (sec)	Temp. (°C)	Gap Height (μm)
1	Gap*	10	110	0.001
2	Bake	80	110	Full contact

*Gap used to imitate slow heating of substrate.
Use 165 sec bake if gap function not available.

Develop: Constant Spray at 27°C

Step	Event	Time (sec)	Speed (rpm)	Accel (krpm/sec)
1	Spray*	260	250	50
2	Rinse	20	300	50
3	Dry	15	4000	50

*140 ml of developer per min



AZ® P4000 Thick Film Photoresist

Modeling Parameters (AZ® P4000 Photoresist at 435 nm)

Refractive Index

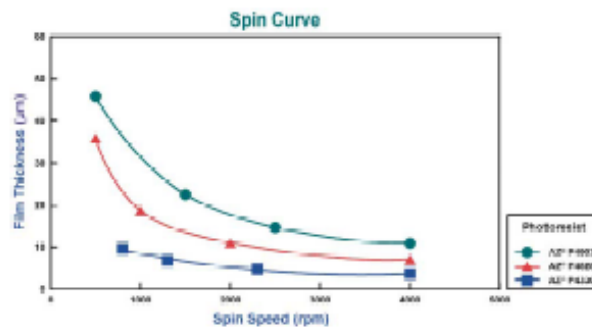
Unbleached		Bleached	
n	1.6963	n	1.6796
k	0.0150	k	0.0100

Cauchy Coefficients

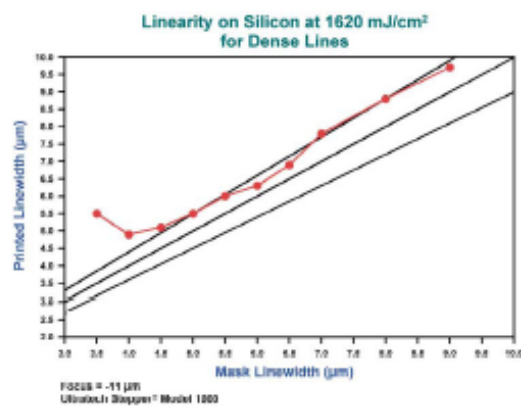
	A	B	C
Unbleached	1.6154	$1.0340 \times 10^{-2} \mu\text{m}^2$	$8.16 \times 10^{-4} \mu\text{m}^4$
Bleached	1.6207	$2.9136 \times 10^{-3} \mu\text{m}^2$	$2.78 \times 10^{-3} \mu\text{m}^4$

Dill Parameters

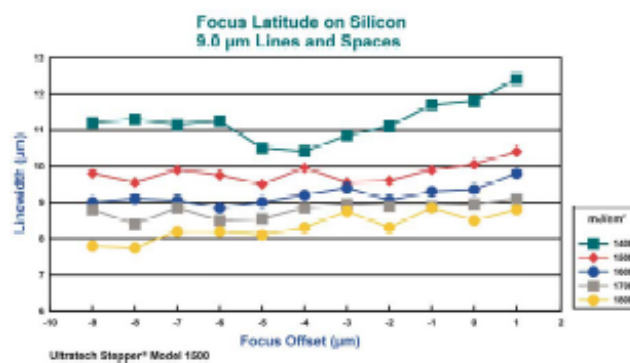
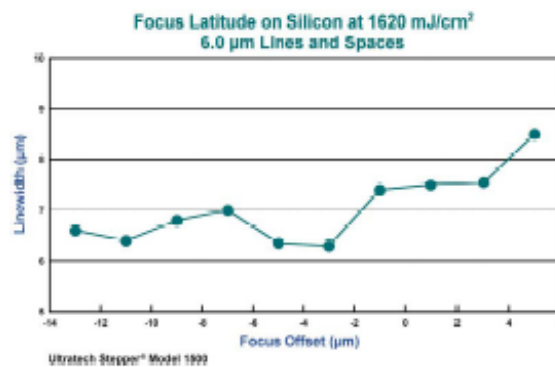
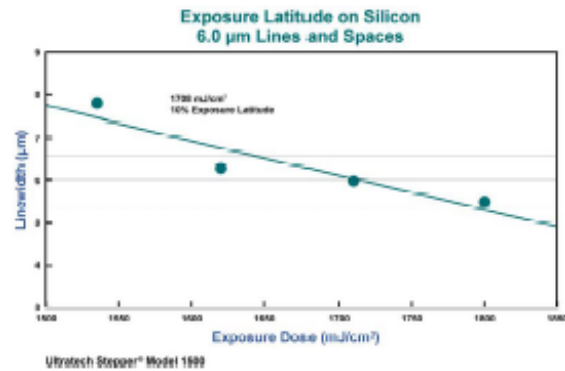
A	B	C
$0.3697 \mu\text{m}^{-1}$	$0.0243 \mu\text{m}^{-1}$	$0.0203 \text{ cm}^2/\text{mJ}$



Performance of AZ® P4620 Photoresist



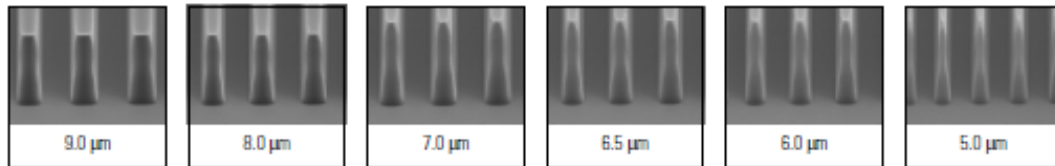
Performance (continued)



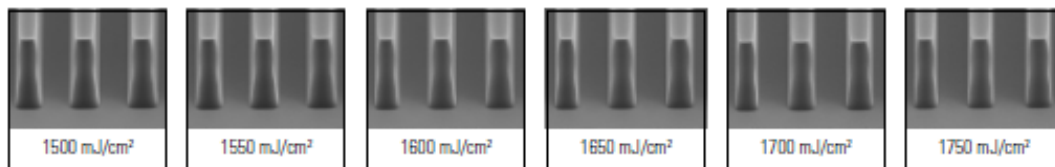
Performance (continued)

24 μm film thickness, double coat/bake at 110°C, on Ultratech Stepper® model 1500, AZ® 400K 1:4
Developer, 260 sec spray

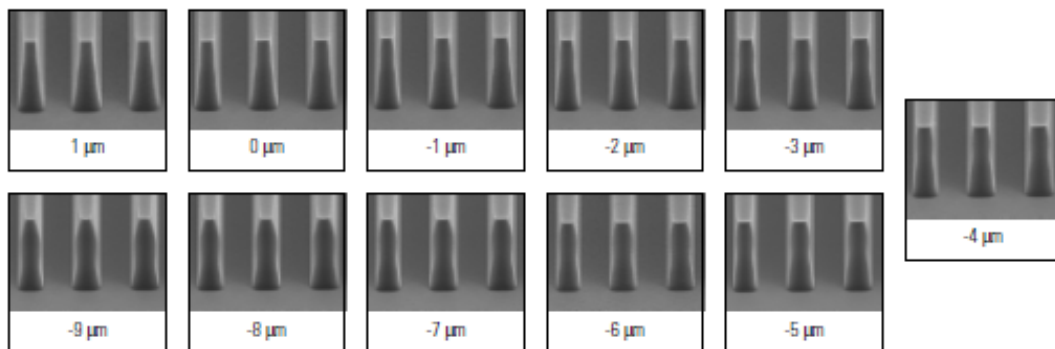
Linearity (1600 mJ/cm²)



Exposure Latitude (9 μm lines and spaces)



Focus Latitude (9 μm lines and spaces, 1600 mJ/cm²)





AZ® P4000 Thick Film Photoresist

Companion Products

Adhesion Promoter

AZ® Adhesion Promoter is highly purified HMDS recommended to promote adhesion of photoresist to semiconductor wafers.

Edge Bead Removers

AZ® EBR 70/30 edge bead remover and AZ EBR solvent are recommended for AZ® P4000 photoresist for both front- and back-side edge bead removal.

Developers

AZ® 400K series and AZ 421K developers are recommended for thick films of AZ P4000 photoresists. These developers may be used for both spray and immersion processes. AZ 400K is a buffered potassium-based developer that provides the process latitude associated with inorganic developers while minimizing risk associated with mobile ion contamination. AZ 421K developer is unbuffered. An alternative sodium-based developer, AZ Developer, has a very low etch rate on aluminum and can also be used with AZ P4000 photoresist. Developer bulletins with additional processing details are available.

Strippers

AZ® 400T and 300T strippers are recommended for removal of AZ P4000 photoresist. AZ 400K developer concentrate can also be used for stripping when a corrosion resistant substrate is used. Using this developer for stripping provides the added benefit of an all-aqueous (organic-solvent-free) system. This results in a quantitative reduction of organic residues as evidenced by the hydrophilic surface obtained after resist removal. Gold surfaces are an exception: they are not hydrophilic after stripping because they are hydrophobic by nature.

Solvent Safety

AZ P4000 photoresist is formulated with propylene glycol monomethyl ether acetate (PGMEA) solvent, which is patented for use in photoresists by Clariant AG (U.S. patent number 4,550,069).

Equipment Compatibility

AZ P4000 photoresist is compatible with all commercially available wafer and photomask processing equipment. Recommended materials of construction include stainless steel, glass, ceramic, PTFE, polypropylene, and high density polyethylene.

Storage

Keep in sealed original containers away from oxidants, sparks, and open flames. Refrigerate until use, and bring to ambient temperature prior to use. Protect from light and heat. Empty container may contain harmful residue and vapors.

Handling Precautions/First Aid

Refer to the current Material Safety Data Sheet (MSDS) for detailed information prior to handling.

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Clariant (Taiwan) Co., Ltd.
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Page 6 of 6 AZ® P4000 Thick Film Photoresist ■

APPENDIX B – MICROCHEM SU-8 2000 SERIES

PERMANENT EPOXY NEGATIVE

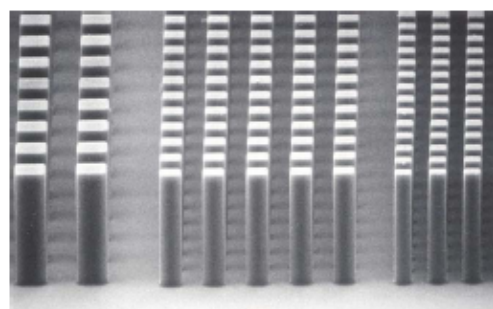
PHOTORESIST DATA SHEET

SU-8 2000

Permanent Epoxy Negative Photoresist

PROCESSING GUIDELINES FOR: SU-8 2025, SU-8 2035, SU-8 2050 and SU-8 2075

SU-8 2000 is a high contrast, epoxy based photoresist designed for micromachining and other microelectronic applications, where a thick, chemically and thermally stable image is desired. SU-8 2000 is an improved formulation of SU-8, which has been widely used by MEMS producers for many years. The use of a faster drying, more polar solvent system results in improved coating quality and increases process throughput. SU-8 2000 is available in twelve standard viscosities. Film thicknesses of 0.5 to >200 microns can be achieved with a single coat process. The exposed and subsequently thermally cross-linked portions of the film are rendered insoluble to liquid developers. SU-8 2000 has excellent imaging characteristics and is capable of producing very high aspect ratio structures. SU-8 2000 has very high optical transmission above 360 nm, which makes it ideally suited for imaging near vertical sidewalls in very thick films. SU-8 2000 is best suited for permanent applications where it is imaged, cured and left on the device.



10 um features, 50 um SU-8 2000 coating

SU-8 2000 Features

- High aspect ratio imaging
- 0.5 to > 200 μm film thickness in a single coat
- Improved coating properties
- Faster drying for increased throughput
- Near UV (350-400 nm) processing
- Vertical sidewalls

Processing Guidelines

SU-8 2000 photoresist is most commonly exposed with conventional UV (350-400 nm) radiation, although i-line (365 nm) is the recommended wavelength. SU-8 2000 may also be exposed with e-beam or x-ray radiation. Upon exposure, cross-linking proceeds in two steps (1) formation of a strong acid during the exposure step, followed by (2) acid-catalyzed, thermally driven epoxy cross-linking during the post exposure bake (PEB) step. A normal process is: spin coat, soft bake, expose, PEB, followed by develop. A controlled hard bake is recommended to further cross-link the imaged SU-8 2000 structures when they will remain as part of the device. The entire process should be optimized for the specific application. The baseline information presented here is meant to be used as a starting point for determining a process.

Process Flow



Table 1. SU-8 2000 Viscosity

SU-8 2000	% Solids	Viscosity (cSt)	Density (g/ml)
2025	68.55	4500	1.219
2035	69.95	7000	1.227
2050	71.65	12900	1.233
2075	73.45	22000	1.236

Substrate Preparation

To obtain maximum process reliability, substrates should be clean and dry prior to applying SU-8 2000 resist. For best results, substrates should be cleaned with a piranha wet etch (using H_2SO_4 & H_2O_2) followed by a de-ionized water rinse. Substrates may also be cleaned using reactive ion etching (RIE) or any barrel asher supplied with oxygen. Adhesion promoters are typically not required. For applications that include electroplating, a pre-treatment of the substrate with MCC Primer 80/20 (HMDS) is recommended.

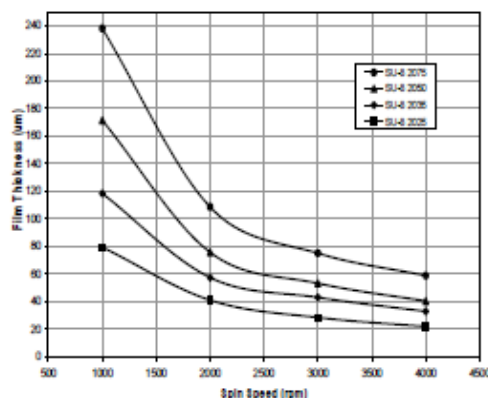
Coat

SU-8 2000 resists are available in twelve standard viscosities. This processing guideline document addresses four products: SU-8 2025, SU-8 2035, SU-8 2050 and SU-8 2075. Figure 1. provides the information required to select the appropriate SU-8 2000 resist and spin conditions to achieve the desired film thickness.

Recommended Program

- 1.) Dispense 1ml of resist for each inch (25mm) of substrate diameter.
- 2.) Spin at 500 rpm for 5-10 seconds with acceleration of 100 rpm/second.
- 3.) Spin at 2000 rpm for 30 seconds with acceleration of 300 rpm/second.

Figure 1. SU-8 2000 Spin Speed versus Thickness



Edge Bead Removal (EBR)

During the spin coat process step, a build up of photoresist may occur on the edge of the substrate. In order to minimize contamination of the hotplate, this thick bead should be removed. This can be accomplished by using a small stream of solvent (MicroChem's EBR PG) at the edge of the wafer either at the top or from the bottom. Most automated spin coaters now have this feature and can be programmed to do this automatically.

By removing any edge bead, the photomask can be placed into close contact with the wafer, resulting in improved resolution and aspect ratio.

Soft Bake

A level hotplate with good thermal control and uniformity is recommended for use during the Soft Bake step of the process. Convection ovens are not recommended. During convection oven baking, a skin may form on the resist. This skin can inhibit the evolution of solvent, resulting in incomplete drying of the film and/or extended bake times. Table 2. shows the recommended Soft Bake temperatures and times for the various SU-8 2000 products at selected film thicknesses.

Note: To optimize the baking times/conditions, remove the wafer from the hotplate after the prescribed time and allow it to cool to room temperature. Then, return the wafer to the hotplate. If the film 'wrinkles', leave the wafer on the hotplate for a few more minutes. Repeat the cool-down and heat-up cycle until 'wrinkles' are no longer seen in the film.

THICKNESS microns	SOFT BAKE TIMES	
	(65°C) minutes	(95°C) minutes
25 - 40	0 - 3	5 - 6
45 - 80	0 - 3	6 - 9
85 - 110	5	10 - 20
115 - 150	5	20 - 30
160 - 225	7	30 - 45

Table 2. Soft Bake Times

Optical Parameters

The dispersion curve and Cauchy coefficients are shown in Figure 3. This information is useful for film thickness measurements based on ellipsometry and other optical measurements.

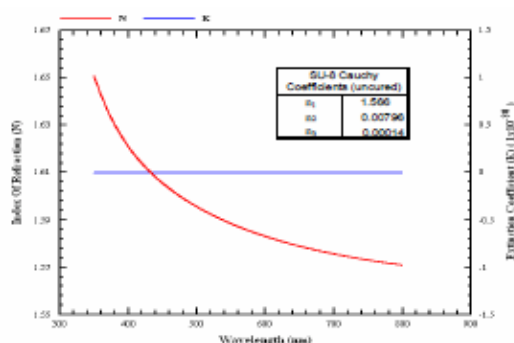


Figure 3. Cauchy Coefficients

Exposure

To obtain vertical sidewalls in the SU-8 2000 resist, we recommend the use of a long pass filter to eliminate UV radiation below 350 nm. With the recommended filter (PL-380-LP) from Omega Optical (www.omegafilters.com) or Asahi Technoglass filters V-42 plus UV-D35 (www.atgc.co.jp), an increase in exposure time of approximately 40% is required to reach the optimum exposure dose.

Note: With optimal exposure, a visible latent image will be seen in the film within 5-15 seconds after being placed on the PEB hotplate and not before. An exposure matrix experiment should be performed to determine the optimum dosage.

THICKNESS microns	EXPOSURE ENERGY mJ/cm ²
25 - 40	150 - 160
45 - 80	150 - 215
85 - 110	215 - 240
115 - 150	240 - 260
160 - 225	260 - 350

Table 3. Exposure Dose

	RELATIVE DOSE
Silicon	1X
Glass	1.5X
Pyrex	1.5X
Indium Tin Oxide	1.5X
Silicon Nitride	1.5 - 2X
Gold	1.5 - 2X
Aluminum	1.5 - 2X
Nickel Iron	1.5 - 2X
Copper	1.5 - 2X
Nickel	1.5 - 2X
Titanium	1.5 - 2X

Table 4. Exposure Doses for Various Substrates

Post Exposure Bake (PEB)

PEB should take place directly after exposure. Table 5. shows the recommended times and temperatures

Note: After 1 minute of PEB at 95°C, an image of the mask should be visible in the SU-8 2000 photoresist coating. If no visible latent image is seen during or after PEB this means that there was insufficient exposure, heating or both.

THICKNESS microns	PEB TIME (65°C)* minutes	PEB TIME (95°C) minutes
25 - 40	1	5 - 6
45 - 80	1 - 2	6 - 7
85 - 110	2 - 5	8 - 10
115 - 150	5	10 - 12
160 - 225	5	12 - 15

* Optional step for stress reduction

Table 5. Post Exposure Bake Times

Development

SU-8 2000 photoresist has been designed for use in immersion, spray or spray-puddle processes with MicroChem's SU-8 developer. Other solvent based developers such as ethyl lactate and diacetone alcohol may also be used. Strong agitation is recommended when developing high aspect ratio and/or thick film structures. The recommended development times for immersion processes are given in Table 6. These development times are approximate, since actual dissolution rates can vary widely as a function of agitation

Note: The use of an ultrasonic or megasonic bath may be helpful when developing out via or hole patterns or structures with tight pitch.

THICKNESS microns	DEVELOPMENT TIME minutes
25 - 40	4 - 5
45 - 75	5 - 7
80 - 110	7 - 10
115 - 150	10 - 15
160 - 225	15 - 17

Table 6. Development Times for SU-8 Developer

Rinse and Dry

When using SU-8 developer, spray and wash the developed image with fresh solution for approximately 10 seconds, followed by a second spray/wash with Isopropyl Alcohol (IPA) for another 10 seconds. Air dry with filtered, pressurized air or nitrogen.

Note: A white film produced during IPA rinse is an indication of underdevelopment of the unexposed photoresist. Simply immerse or spray the substrate with additional SU-8 developer to remove the white film and complete the development process. Repeat the rinse step.

The use of an ultrasonic or megasonic bath will energize the solvent and allow for more effective development of the unexposed resist.

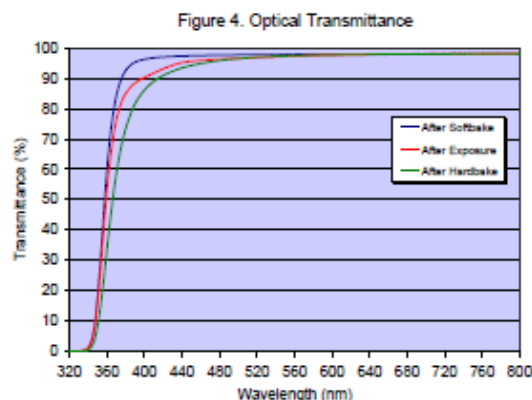
Physical Properties

(Approximate values)

Adhesion Strength (mPa) Silicon/Glass/Glass & HMDS	38/35/35
Glass Transition Temperature (T _g °C), tan δ peak	210
Thermal Stability (°C @ 5% wt. loss)	315
Thermal Conductivity (W/mK)	0.3
Coeff. of Thermal Expansion (CTE ppm)	52
Tensile Strength (Mpa)	60
Elongation at break (εb %)	6.5
Young's Modulus (Gpa)	2.0
Dielectric Constant @ 10MHz	3.2
Water Absorption (% 85°C/85 RH)	0.65

Table 7. Physical Properties

Optical Properties



Process conditions for Figure 4.

Softbake: 5 minutes at 95°C

Exposure: 180 mJ/cm²

Hardbake: 30 minutes at 300°C

Hard Bake (cure)

SU-8 2000 has good mechanical properties. However, for applications where the imaged resist is to be left as part of the final device, a hard bake can be incorporated into the process. This is generally only required if the final device or part is to be subject to thermal processing during regular operation. A hard bake or final cure step is added to ensure that SU-8 2000 properties do not change in actual use. SU-8 2000 is a thermal resin and as such its properties can continue to change when exposed to a higher temperature than previously encountered. We recommend using a final bake temperature 10°C higher than the maximum expected device operating temperature. Depending on the degree of cure required, a bake temperature in the range of 150°C to 250°C and for a time between 5 and 30 minutes is typically used.

Note: The hard bake step is also useful for annealing any surface cracks that may be evident after development. The recommended step is to bake at 150°C for a couple of minutes. This applies to all film thicknesses.



www.microchem.com

Removal

SU-8 2000 has been designed as a permanent, highly cross-linked epoxy material and it is extremely difficult to remove it with conventional solvent based resist strippers. MicroChem's Remover PG will swell and lift off minimally cross-linked SU-8 2000. However, if OmniCoat (30-100 nm) has been applied, immersion in Remover PG can effect a clean and thorough Lift-Off of the SU-8 2000 material. Fully cured or hard baked SU-8 2000 cannot be removed without the use of OmniCoat.

To remove minimally cross-linked SU-8 2000, or when using OmniCoat: Heat the Remover PG bath to 50-80°C and immerse the substrates for 30-90 minutes. Actual strip time will depend on resist thickness and cross-link density. For more information on MicroChem OmniCoat and Remover PG please see the relevant product data sheets.

To re-work fully cross-linked SU-8 2000: Wafers can be stripped using oxidizing acid solutions such as piranha etch, plasma ash, RIE, laser ablation and pyrolysis.

Plasma Removal

RIE 200W, 80 sccm O₂, 8 sccm CF₄, 100mTorr, 10°C

Storage

Store SU-8 2000 resists upright and in tightly closed containers in a cool, dry environment away from direct sunlight at a temperature of 40-70°F (4-21°C). Store away from light, acids, heat and sources of ignition. Shelf life is thirteen months from date of manufacture.

Disposal

SU-8 2000 resists may be included with other waste containing similar organic solvents to be discarded for destruction or reclaim in accordance with local state and federal regulations. It is the responsibility of the customer to ensure the disposal of SU-8 2000 resists and residues made in observance all federal, state, and local environmental regulations.

Environmental, Health and Safety

Consult the product Material Safety Data Sheet before working with SU-8 2000 resists. Handle with care. Wear chemical goggles, chemical gloves and suitable protective clothing when handling SU-8 2000 resists. Do not get into eyes, or onto skin or clothing. Use with adequate ventilation to avoid breathing vapors or mist. In case of contact with skin, wash affected area with soap and water. In case of contact with eyes, rinse immediately with water and flush for 15 minutes lifting eyelids frequently. Get emergency medical assistance.

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